

Supporting Information

Defluorinative Alkylation of 1-Trifluoromethyl Alkenes with Alkyl radicals Derived from Visible-Light Induced Deoxygenation of Xanthate Salts: Synthesis of gem-Difluoroalkenes

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1. General Information.

All new compounds were fully characterized. NMR spectra were recorded on JNM-ECZ400S/L1 and calibrated using residual undeuterated solvent CDCl₃, DMSO-D6 (¹H NMR spectra: CDCl₃: 7.26 ppm, DMSO-D6: 2.50 ppm; ¹³C NMR spectra: CDCl₃: 77.00 ppm, DMSO-D6: 39.50 ppm). Mass spectra were conducted at Bruker MTQ III q-TOF (ESI) Mass Spectrometer. Cyclic voltammetry (CV) experiments were used on CHI660E. Measurements of UV-Vis absorption spectra were made on SHIMADZU UV-3600Plus. Anhydrous solvents, such as Dichloromethane (DCM), *N,N*-Dimethylacetamide (DMA), *N,N*-Dimethylformamide (DMF), Dimethyl sulfoxide (DMSO), Tetrahydrofuran (THF), Ethyl acetate (EA), Methanol (CH₃OH), Acetonitrile (MeCN), Dichloroethane (DCE), *N*-Methyl-2-pyrrolidone (NMP) were purchased from Adamas. Anhydrous acetone was obtained by laboratory refining. Flash column chromatography was carried out using silica gel (General-Reagent, AR, 200-300 mesh, for column chromatography). All reactions were carried out in dried 20 mL vial under Nitrogen. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. TEA and PE are the abbreviation of triethylamine and petroleum ether.

Photoreaction setup:

All Photoredox reactions were subjected to irradiation from double 40W Kessil PR160L blue LED bulbs (456 nm), with the reaction tube placed approximately ~ 5 cm from the bulbs and using a fan to keep at room temperature (Figure S1).

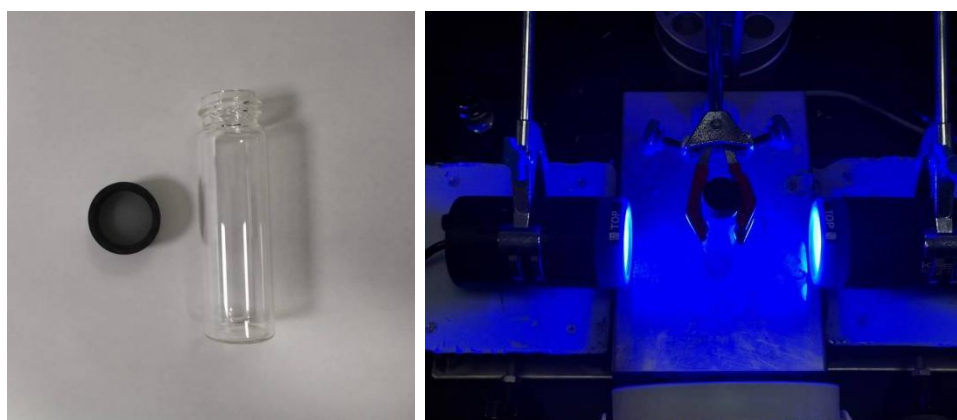


Figure S1. An over-dried 20 mL vial (left). Photoreaction setup (right).

2. Optimization of reaction conditions

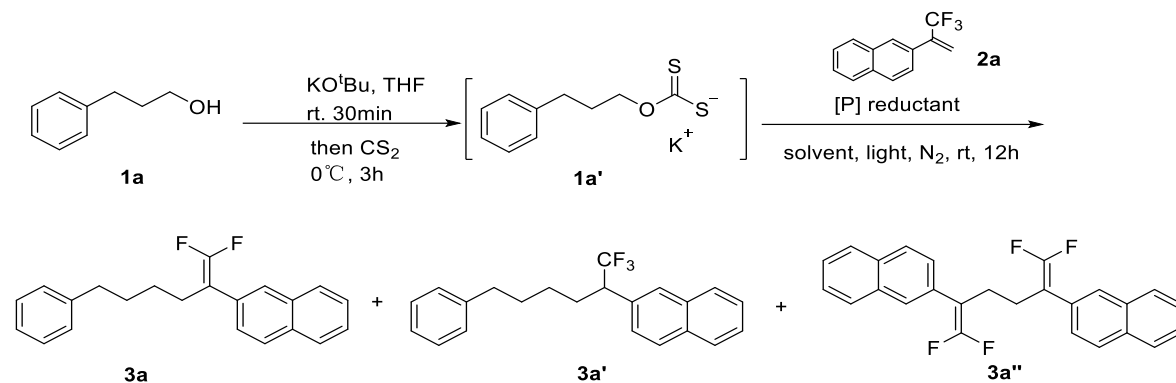


Table S1. Exploration of reductants^a

Entry	Reductant	Yield of 3a (%)	Yield of 3a' (%)	Yield of 3a'' (%)	Recovery of 2a (%)
1	PPh ₃	2	0	32	30
2	P ⁿ Bu ₃	60	0	2	13
3	P(OEt) ₃	3	0	28	40
4	P ^t Bu ₃	60	0.33	7.5	1
5	1,4-bis(dicyclohexylphosphino)butane	74	0	3.5	13
6	PCy ₃	75	0	5	13
7	Ph ₂ PCy	31	0	10.5	52
8	PhPCy₂	80	0	3.5	10

^aReaction conditions: **1a'** (0.1 mmol, 1 eq), **2a** (0.12 mmol, 1.2 eq), DMA (2 mL), Reductant (0.12 mmol, 1.2 eq), Kessil PR160L (456 nm), I = 25%, rt, 12 h, N₂ atmosphere. Yields were determined by ¹⁹F NMR with 1, 4-Difluorobenzene (9.8 μL, 0.1 mmol) as an internal standard.

Table S2. Exploration of solvents^a

Entry	Solvent	Yield of 3a (%)	Yield of 3a' (%)	Yield of 3a'' (%)	Recovery of 2a (%)
9	DMA (2 mL)	80	0	3.5	10
10	DMF (2 mL)	80	0	3	11
11	DMSO (2 mL)	82	0	5	9
12	THF (2 mL)	10	0	0	85
13	EA (2 mL)	11	0	0	92
14	Acetone (2 mL)	83	1.3	1	18
15	CH ₃ OH (2 mL)	0	1.7	0	95
16	CH ₃ CN (2 mL)	78	0	2	44
17	DCE (2 mL)	0	0	5	91
18	NMP (2 mL)	75	0.33	3.5	7
19	DMSO/DCM=3/1 (2 mL)	0	0	2	91
20	CH ₃ CN/DCM=3/1 (2 mL)	10	0	1	93
21	DMSO/CH ₃ CN=19/1 (2 mL)	78	0	7	5
22	DMSO/CH ₃ CN=9/1 (2 mL)	86	0	5	8
23	DMSO/CH ₃ CN=4/1 (2 mL)	82	0	4	10
24	DMSO/CH ₃ CN=3/1 (2 mL)	83	0	4	9
25	DMSO/CH ₃ CN=1/1 (2 mL)	79	0	3.5	15
26	DMSO/CH ₃ CN=1/3 (2 mL)	73	0	2	21
27	DMSO/CH ₃ CN=1/4 (2 mL)	79	0	2	16
28	DMSO/CH ₃ CN=1/9 (2 mL)	80	0	1.5	16
29	Acetone/CH ₃ CN=19/1 (2 mL)	86	1.3	3	26
30	Acetone/CH₃CN=9/1 (2 mL)	90	1	3	22
31	Acetone/CH ₃ CN=4/1 (2 mL)	85	0.67	3	25
32	Acetone/CH ₃ CN=3/1 (2 mL)	87	1	3	25
33	Acetone/CH ₃ CN=1/1 (2 mL)	87	0.67	2.5	21
34	Acetone/CH ₃ CN=1/3 (2 mL)	77	0.33	1.5	19
35	Acetone/CH ₃ CN=1/4 (2 mL)	81	0.33	2	24
36	Acetone/MeCN=9/1 (1 mL)	76	1	3	23
37	Acetone/MeCN=9/1 (4 mL)	81	1.7	2	19

^aReaction conditions: **1a'** (0.1 mmol, 1 eq), **2a** (0.12 mmol, 1.2 eq), PhPCy₂ (0.12 mmol, 1.2 eq), Solvent, Kessil PR160L (456 nm), I = 25%, rt, 12 h, N₂ atmosphere. Yields were determined by

¹⁹F NMR with 1, 4-Difluorobenzene (9.8 μL, 0.1 mmol) as an internal standard.

Table S3. Exploration of light sources^a

Entry	Light (nm)	Intensity (%)	Yield of 3a (%)	Yield of 3a' (%)	Yield of 3a'' (%)	Recovery of 2a (%)
38	390	25	78	1.3	4	8
39	427	25	83	1	2.5	16
40	440	25	83	1	3	23
41	456	25	90	1	3	22
42	456	50	83	1	3	13
43	456	75	84	1.3	2.5	18
44	456	100	81	1.3	3	13

^aReaction conditions: **1a'** (0.1 mmol, 1 eq), **2a** (0.12 mmol, 1.2 eq), PhPCy₂ (0.12 mmol, 1.2 eq), Acetone/CH₃CN = (1.8 mL/0.2mL), Kessil PR160L (λ nm), I (%), rt, 12 h, N₂ atmosphere. Yields were determined by ¹⁹F NMR with 1, 4-Difluorobenzene (9.8 μL, 0.1 mmol) as an internal standard.

Table S4. Exploration of olefin and reductant contents^a

Entry	Olefin content (eq)	Reductant content (eq)	Yield of 3a (%)	Yield of 3a' (%)	Yield of 3a'' (%)	Recovery of 2a (%)
45	1.05	1.2	75	0.33	2	8
46	1.1	1.2	79	0.33	2	13
47	1.2	1.2	90	1	3	22
48	1.2	1.1	85	1.3	2	24
49	1.2	1.5	84	1.3	2.5	21
50	1.2	2.0	82	1.3	2.5	21

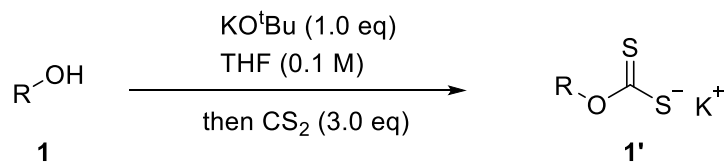
^aReaction conditions: **1a'** (0.1 mmol, 1 eq), **2a**, PhPCy₂, Acetone/CH₃CN = (1.8 mL/0.2 mL), Kessil PR160L (456 nm), I = 25%, rt, 12 h, N₂ atmosphere. Yields were determined by ¹⁹F NMR with 1, 4-Difluorobenzene (9.8 μL, 0.1 mmol) as an internal standard.

Table S5. Exploration of one-pot procedure and bases^a

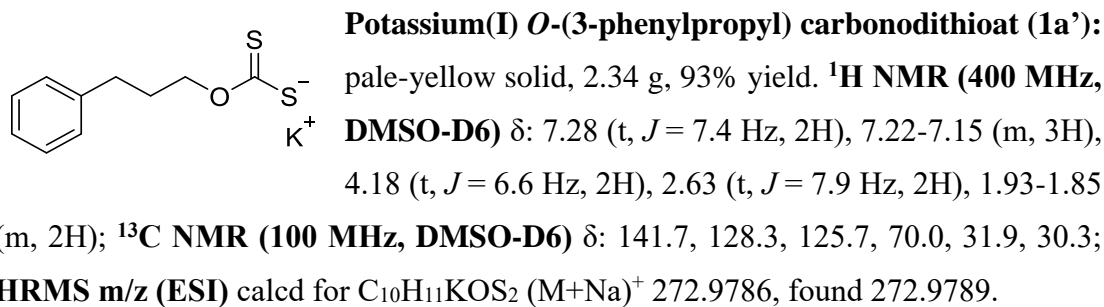
Entry	Base	Yield of 3a (%)	Yield of 3a' (%)	Yield of 3a'' (%)	Recovery of 2a (%)
51	KO ^t Bu (1.0 eq)	78	1.2	2	18
52	KO ^t Bu (1.05 eq)	68	1.2	2	28
53	KO^tBu (1.1 eq)	87	1.8	2.8	19
54	NaH (1.1 eq)	11	2.5	10	38
55	NaOH (1.1 eq)	2	3.3	13	42
56	KOH (1.1 eq)	19	3.2	12	27
57	KHDMS (1.1 eq)	74	0.4	2.5	23
58 ^b	KO ^t Bu (1.1 eq)	0	0	0	99
59 ^c	KO ^t Bu (1.1 eq)	0	0	0	95

^aReaction conditions: **1a** (0.1 mmol), base, CS₂ (0.3 mmol), THF (1 mL), 3 h. After the solvent was removed in vacuo, then **2a** (0.12 mmol), PPhCy₂ (0.12 mmol), acetone/CH₃CN = 9:1 (2 mL), Kessil PR160L (456 nm), I = 25%, rt, 12 h, N₂ atmosphere. ^bno light. ^cno reductant. Yields were determined by ¹⁹F NMR with 1, 4-Difluorobenzene (9.8 μL, 0.1 mmol) as an internal standard.

3. General Procedure for Synthesis of Xanthate Salts

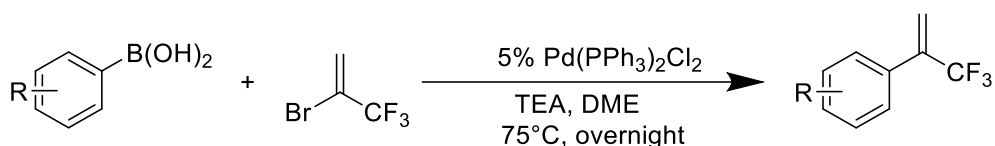


In a N₂-filled glovebox, 250 mL round-bottom flask equipped with a magnetic stir bar was charged sequentially with alcohol **1** (10.0 mmol), **KO^tBu** (1.12 g, 10.0 mmol), and **dry THF** (100 mL). The flask was sealed with a septum cap and transferred out of the glovebox, and a balloon was attached. The reaction mixture was stirred at room temperature for 30 minutes, followed by the addition of **CS₂** (1.8 mL, 30.0 mmol) via microsyringe at 0°C and continued to be stirred for 3 hours at 0°C before removing the solvent under reduced pressure. The solid was washed with Et₂O (2 × 50 mL), and dried in vacuo to afford the desired product.¹



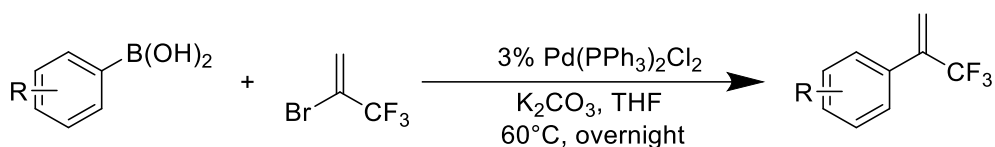
4. Preparation of Trifluoromethyl Alkenes

Method A:²



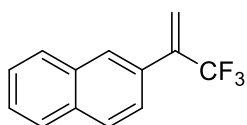
In a Schlenk tube equipped with stir bar, arylboronic acids (1.5 equiv.), $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ (5 mol%) were added. The vessel was evacuated and filled with nitrogen, then TEA (8.0 equiv.), DME (0.33 M) and H_2O (0.67 M) were added. After the addition of 2-bromo-3,3,3-trifluoropropene (1.0 equiv.), the solution was stirred at 75 °C overnight (TLC tracking detection). The mixture was purified by column chromatography to afford the corresponding trifluoromethyl alkenes. (Note: the products might be volatile under high vacuum).

Method B:²

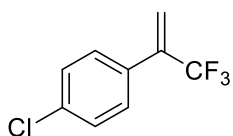


In a Schlenk tube equipped with stir bar, arylboronic acids (1.0 equiv.), $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ (3 mol%) and K_2CO_3 (4.0 equiv.) were added. The vessel was evacuated and filled with nitrogen, then THF (0.33 M) and H_2O (0.5 M) were added. After the

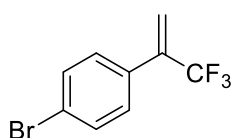
addition of 2-bromo-3,3,3-trifluoropropene (2.0 equiv.), the solution was stirred at 60 °C overnight (TLC tracking detection). The mixture was purified by column chromatography to afford the corresponding trifluoromethyl alkenes. (Note: the products might be volatile under high vacuum).



2-(3,3,3-trifluoroprop-1-en-2-yl)naphthalene (2a) : According to Method A, the reaction was carried out with the corresponding arylboronic acid (5.16 g, 30.00 mmol), Pd(PPh₃)₂Cl₂ (701.9 mg, 1.02 mmol), TEA (8 mL, 142.50 mmol), DME (60 mL) and H₂O (30 mL), 2-bromo-3,3,3-trifluoropropene (2.08 mL, 20.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 4.09 g (92% yield) of **2a** as a white solid: ¹H NMR (400 MHz, CDCl₃) δ: 7.94-7.85 (m, 4H), 7.58-7.51 (m, 3H), 6.05 (s, 1H), 5.91 (s, 1H). All data are in accordance with the literature.²

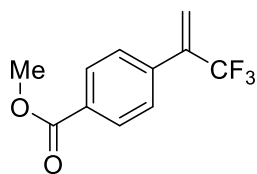


1-Chloro-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene (2b) : According to Method A, the reaction was carried out with the corresponding arylboronic acid (1.88 g, 12.00 mmol), Pd(PPh₃)₂Cl₂ (285.7 mg, 0.41 mmol), TEA (8 mL, 57.00 mmol), DME (24 mL) and H₂O (12 mL), 2-bromo-3,3,3-trifluoropropene (0.83 mL, 8.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 0.64 g (39% yield) of **2b** as a yellow oil: ¹H NMR (400 MHz, CDCl₃) δ: 7.40-7.35 (m, 4H), 5.98 (s, 1H), 5.77 (s, 1H). All data are in accordance with the literature.²



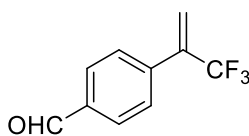
1-Bromo-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene (2c) : According to Method A, the reaction was carried out with the corresponding arylboronic acid (2.41 g, 12.00 mmol), Pd(PPh₃)₂Cl₂ (285.7 mg, 0.41 mmol), TEA (8 mL, 57.00 mmol), DME (24 mL) and H₂O (12 mL), 2-bromo-3,3,3-trifluoropropene (0.83 mL, 8.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 0.81 g (41% yield) of **2c** as a white solid: ¹H NMR (400 MHz, CDCl₃)

δ : 7.52 (d, J = 8.4 Hz, 2H), 7.32 (d, J = 8.6 Hz, 2H), 5.98 (s, 1H), 5.78 (s, 1H). All data are in accordance with the literature.²



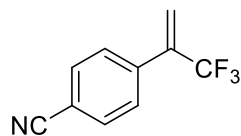
Methyl 4-(3,3,3-trifluoroprop-1-en-2-yl)benzoate (2d) :

According to Method A, the reaction was carried out with the corresponding arylboronic acid (2.16 g, 12.00 mmol), Pd(PPh₃)₂Cl₂ (285.7 mg, 0.41 mmol), TEA (8 mL, 57.00 mmol), DME (24 mL) and H₂O (12 mL), 2-bromo-3,3,3-trifluoropropene (0.83 mL, 8.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE: DCM=3:1) to afford 1.04 g (57% yield) of **2d** as a yellow oil: ¹H NMR (400 MHz, CDCl₃) δ : 8.05 (d, J = 8.3 Hz, 2H), 7.53 (d, J = 8.6 Hz, 2H), 6.05 (s, 1H), 5.87 (s, 1H), 3.94 (s, 1H). All data are in accordance with the literature.²



4-(3,3,3-trifluoroprop-1-en-2-yl)benzaldehyde (2e) :

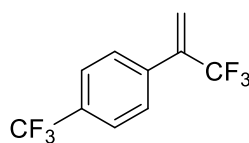
According to Method A, the reaction was carried out with the corresponding arylboronic acid (1.82 g, 12.00 mmol), Pd(PPh₃)₂Cl₂ (285.7 mg, 0.41 mmol), TEA (8 mL, 57.00 mmol), DME (24 mL) and H₂O (12 mL), 2-bromo-3,3,3-trifluoropropene (0.83 mL, 8.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 0.67 g (42% yield) of **2e** as a yellow oil: ¹H NMR (400 MHz, CDCl₃) δ : 10.05 (s, 1H), 7.92 (d, J = 8.4 Hz, 2H), 7.64 (d, J = 8.4 Hz, 1H), 6.09 (s, 1H), 5.90 (s, 1H). All data are in accordance with the literature.³



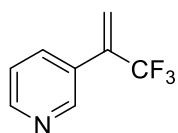
3-(3,3,3-Trifluoroprop-1-en-2-yl)benzonitrile (2f) :

According to Method A, the reaction was carried out with the corresponding arylboronic acid (1.76 g, 12.00 mmol), Pd(PPh₃)₂Cl₂ (285.7 mg, 0.41 mmol), TEA (8 mL, 57.00 mmol), DME (24 mL) and H₂O (12 mL), 2-bromo-3,3,3-trifluoropropene (0.83 mL, 8.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE: DCM=3:1) to afford 1.01 g (64% yield) of **2f** as an orange oil: ¹H NMR (400 MHz, CDCl₃) δ : 7.69 (d, J = 8.4 Hz, 2H), 7.57 (d, J = 7.4 Hz, 1H), 6.11(s, 1H), 5.88 (m, 1H). All data are in

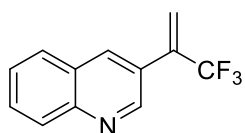
accordance with the literature.²



1-(Trifluoromethyl)-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene (2g) : According to Method A, the reaction was carried out with the corresponding arylboronic acid (2.28 g, 12.00 mmol), Pd(PPh₃)₂Cl₂ (285.7 mg, 0.41 mmol), TEA (8 mL, 57.00 mmol), DME (24 mL) and H₂O (12 mL), 2-bromo-3,3,3-trifluoropropene (0.83 mL, 8.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 0.49 g (26% yield) of **2g** as a yellow oil: ¹H NMR (400 MHz, CDCl₃) δ: 7.66 (d, *J* = 8.2 Hz, 2H), 7.57 (d, *J* = 8.8 Hz, 2H), 6.07 (s, 1H), 5.85 (s, 1H). All data are in accordance with the literature.²

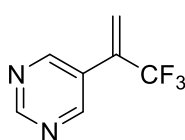


3-(3,3,3-Trifluoroprop-1-en-2-yl)pyridine (2h) : According to Method A, the reaction was carried out with the corresponding arylboronic acid (1.48 g, 12.00 mmol), Pd(PPh₃)₂Cl₂ (285.7 mg, 0.41 mmol), TEA (8 mL, 57.00 mmol), DME (24 mL) and H₂O (12 mL), 2-bromo-3,3,3-trifluoropropene (0.83 mL, 8.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE: DCM=5:1) to afford 0.82 g (59% yield) of **2h** as a brown oil: ¹H NMR (400 MHz, CDCl₃) δ: 8.70 (s, 1H), 8.64 (d, *J* = 4.7 Hz, 1H), 7.78 (d, *J* = 8.7 Hz, 1H), 7.34 (t, *J* = 7.0 Hz, 1H), 6.07 (s, 1H), 5.85 (s, 1H). All data are in accordance with the literature.²

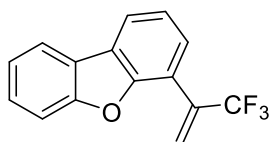


3-(3,3,3-Trifluoroprop-1-en-2-yl)quinoline (2i) : According to Method B, the reaction was carried out with the corresponding arylboronic acid (1.39 g, 8.00 mmol), Pd(PPh₃)₂Cl₂ (168.5 mg, 0.24 mmol), K₂CO₃ (4.42 g, 32.00 mmol), THF (24 mL) and H₂O (16 mL), 2-bromo-3,3,3-trifluoropropene (1.66 mL, 16.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE: EA=10:1) to afford 1.03 g (58% yield) of **2i** as a yellow solid: ¹H NMR (400 MHz, CDCl₃) δ: 8.99 (s, 1H), 8.25 (s, 1H), 8.13 (d, *J* = 8.6 Hz, 1H), 7.87 (d, *J* = 8.0 Hz, 1H), 7.77 (t, *J* = 7.0 Hz, 1H), 7.61 (t, *J* = 7.0 Hz, 1H), 6.17 (s, 1H), 6.00 (s, 1H). All data are in accordance with the

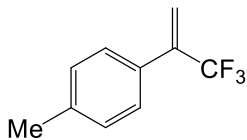
literature.²



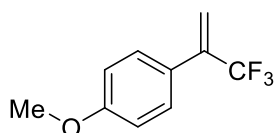
5-(3,3,3-trifluoroprop-1-en-2-yl)pyrimidine (2j) : According to Method A, the reaction was carried out with the corresponding arylboronic acid (1.82 g, 12.00 mmol), Pd(PPh₃)₂Cl₂ (285.7 mg, 0.41 mmol), TEA (8 mL, 57.00 mmol), DME (24 mL) and H₂O (12 mL), 2-bromo-3,3,3-trifluoropropene (0.83 mL, 8.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 0.67 g (48% yield) of **2j** as a yellow oil: ¹H NMR (400 MHz, CDCl₃) δ: 9.25 (s, 1H), 8.83 (s, 2H), 6.17 (s, 1H), 5.93 (s, 1H). All data are in accordance with the literature.³



4-(3,3,3-Trifluoroprop-1-en-2-yl)dibenzo[b,d]furan (2k) : According to Method B, the reaction was carried out with the corresponding arylboronic acid (1.70 g, 8.00 mmol), Pd(PPh₃)₂Cl₂ (168.5 mg, 0.24 mmol), K₂CO₃ (4.42 g, 32.00 mmol), THF (24 mL) and H₂O (12 mL), 2-bromo-3,3,3-trifluoropropene (1.66 mL, 16.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 1.6 g (76% yield) of **2k** as a yellow oil: ¹H NMR (400 MHz, CDCl₃) δ: 7.98 (d, *J* = 7.6 Hz, 2H), 7.60 (d, *J* = 8.3 Hz, 1H), 7.54-7.47 (m, 2H), 7.37 (t, *J* = 7.6 Hz, 2H), 6.37 (t, *J* = 1.25 Hz, 2H). All data are in accordance with the literature.²

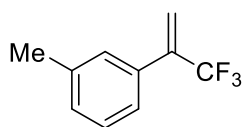


Methyl-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene (2l) : According to Method A, the reaction was carried out with the corresponding arylboronic acid (1.63 g, 12.00 mmol), Pd(PPh₃)₂Cl₂ (285.7 mg, 0.41 mmol), TEA (8 mL, 57.00 mmol), DME (24 mL) and H₂O (12 mL), 2-bromo-3,3,3-trifluoropropene (0.83 mL, 8.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 0.49 g (33% yield) of **2l** as a colorless oil: ¹H NMR (400 MHz, CDCl₃) δ: 7.36 (d, *J* = 7.0 Hz, 2H), 7.20 (d, *J* = 6.5 Hz, 2H), 5.91 (s, 1H), 5.74 (s, 1H), 2.38 (s, 3H). All data are in accordance with the literature.²



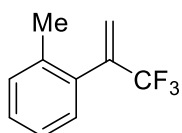
Methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene (2m) :

According to Method A, the reaction was carried out with the corresponding arylboronic acid (1.82 g, 12.00 mmol), Pd(PPh₃)₂Cl₂ (285.7 mg, 0.41 mmol), TEA (8 mL, 57.00 mmol), DME (24 mL) and H₂O (12 mL), 2-bromo-3,3,3-trifluoropropene (0.83 mL, 8.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 0.67 g (41% yield) of **1c** as a yellow oil: ¹H NMR (400 MHz, CDCl₃) δ: 7.40 (d, *J* = 9.0 Hz, 2H), 6.91 (d, *J* = 8.5 Hz, 2H), 5.87 (s, 1H), 5.70 (s, 1H), 3.83 (s, 3H). All data are in accordance with the literature.²



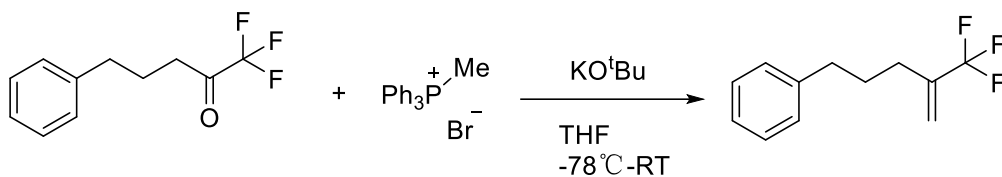
1-methyl-3-(3,3,3-trifluoroprop-1-en-2-yl)benzene (2n) :

According to Method B, the reaction was carried out with the corresponding arylboronic acid (816 mg, 6.00 mmol), Pd(PPh₃)₂Cl₂ (126.0 mg, 0.18 mmol), K₂CO₃ (3.32 g, 24.00 mmol), THF (18 mL) and H₂O (12 mL), 2-bromo-3,3,3-trifluoropropene (1.25 mL, 12.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 850 mg (76% yield) of **2n** as a colorless oil: ¹H NMR (400 MHz, CDCl₃) δ: 7.30-7.24 (m, 3H), 7.20 (d, *J* = 6.7 Hz, 1H), 5.94 (s, 1H), 5.75 (s, 1H), 2.38 (s, 3H). All data are in accordance with the literature.⁴

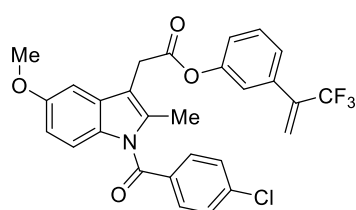
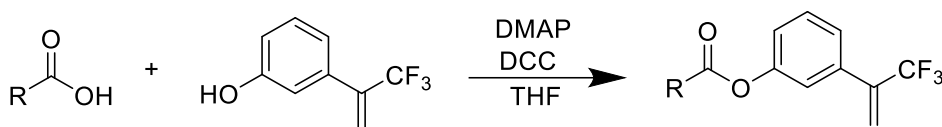


1-methyl-2-(3,3,3-trifluoroprop-1-en-2-yl)benzene (2o):

According to Method B, the reaction was carried out with the corresponding arylboronic acid (816 mg, 6.00 mmol), Pd(PPh₃)₂Cl₂ (126.0 mg, 0.18 mmol), K₂CO₃ (3.32 g, 24.00 mmol), THF (18 mL) and H₂O (12 mL), 2-bromo-3,3,3-trifluoropropene (1.25 mL, 12.00 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 582 mg (52% yield) of **2o** as a colorless oil: ¹H NMR (400 MHz, CDCl₃) δ: 7.30-7.24 (m, 2H), 7.20 (d, *J* = 3.1 Hz, 2H), 6.11 (s, 1H), 5.49 (s, 1H), 2.30 (s, 3H). All data are in accordance with the literature.⁵

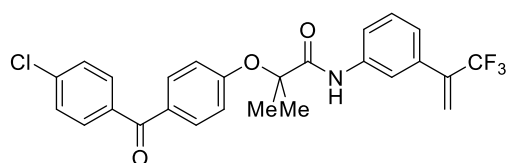
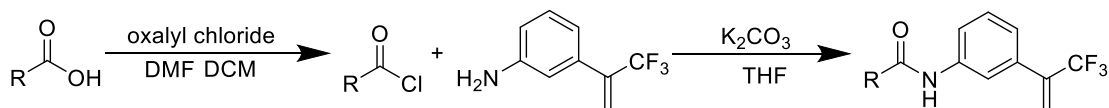


(4-(trifluoromethyl)pent-4-en-1-yl)benzene (2p): In a N₂-filled glovebox, 100 mL round-bottom flask equipped with a magnetic stir bar was charged sequentially with 1,1,1-trifluoro-5-phenyl-2-pentanone (1.30 g, 6.0 mmol, 1.0 equiv.) and **dry THF** (20 mL). Then the mixture was cooled to -78 °C, and dropwise added (4 mL) tetrahydrofuran-dissolved methyltriphenylphosphonium bromide (2.57 g, 7.2 mmol, 1.2 equiv.). The reaction mixture was stirred at room temperature for 1.5 hours. The resultant mixture was treated with NH₄Cl (20 mL). After extraction with methylene chloride, the combined organic phases were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. The mixture was purified by flash column chromatography on silica gel (PE) to afford 878 mg (68% yield) of **2p** as a colorless oil: ¹H NMR (400 MHz, CDCl₃) δ: 7.33-7.26 (m, 2H), 7.22-7.16 (m, 3H), 5.68 (s, 1H), 5.32 (s, 1H), 2.67 (t, *J* = 7.8 Hz, 2H), 2.25 (t, *J* = 7.8 Hz, 2H), 1.90-1.82 (m, 2H). All data are in accordance with the literature.⁶



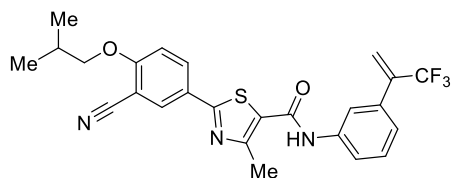
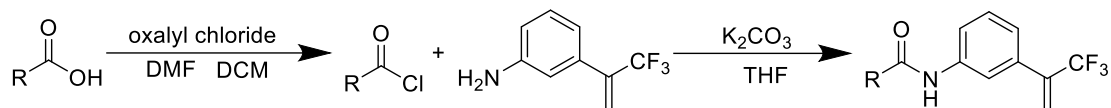
3-(3,3,3-Trifluoroprop-1-en-2-yl)phenyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (2q): To a solution of carboxylic acid (3.26 g, 9.10 mmol, 1.0 equiv.), 4-dimethylaminopyridine (DMAP) (111.2 mg, 0.90 mmol, 0.1 equiv.) and 3-(3,3,3-Trifluoroprop-1-en-2-yl)phenol (1.88 g, 10.00 mmol, 1.1 equiv.) in DMF (22.75 mL), *N,N*-dicyclohexylcarbodiimide (DCC) (2.06 g, 10.00 mmol, 1.1 equiv.) was added. The reaction mixture was stirred at room temperature for 5 hours (TLC tracking detection). The mixture was purified by flash column chromatography on silica gel (PE: DCM=1:5) to afford 3.2 g (67% yield) of **2q** as a yellow oil: ¹H NMR (400 MHz, CDCl₃) δ: 7.68 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.40-7.30 (m, 2H),

7.20 (s, 1H), 7.14-7.11 (m, 1H), 7.09 (d, $J = 2.5$ Hz, 1H), 6.93 (d, $J = 9.0$ Hz, 1H), 6.73 (dd, $J = 9.0$ Hz, $J = 2.5$ Hz, 1H), 5.99 (s, 1H), 5.79 (s, 1H), 3.94 (s, 2H), 3.85 (s, 3H), 2.47 (s, 3H). All data are in accordance with the literature.²



**2-(4-(4-Chlorobenzoyl)phenoxy)-2-methyl
-N-(3-(3,3,3-trifluoroprop-1-en-2-yl)phenyl)
propanamide (2r)** :

To a mixture of Fenofibric acid (1.09 g, 3.00 mmol, 1.0 equiv.) and oxalylchloride (0.51 mL, 6.00 mmol, 2.0 equiv.) in dry CH₂Cl₂ (12 mL) was added dropwise DMF (23.4 μ L, 0.30 mmol, 0.1 equiv.). The reaction mixture was stirred at room temperature for 6 hours. Removal of the solvent in vacuo afforded the desired acid chloride which was used in the next step without further purification. To a mixture of 3-(3,3,3-trifluoroprop-1-en-2-yl)aniline (0.57 g, 3.00 mmol, 1.0 equiv.) and K₂CO₃ (0.42 g, 3.00 mmol, 1.0 equiv.) in dry THF (6 mL) was added dropwise a solution of the freshly prepared acid chloride (3.00 mmol, 1.0 equiv.) in dry THF (6 mL). This mixture was stirred at room temperature for 6 hours before water was added to quench the reaction. The resultant mixture was extracted with EtOAc (3*20 mL). The combined organic phases were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. The resultant crude product was purified by column chromatography on silica gel (PE: EA=10:1) to give the desired trifluoromethyl alkene as a white solid 1.10 g (75% yield). ¹H NMR (400 MHz, CDCl₃) δ : 8.41 (s, 1H), 7.77 (d, $J = 8.8$ Hz, 2H), 7.72-7.70 (m, 3H), 7.59-7.56 (m, 1H), 7.45 (d, $J = 8.6$ Hz, 2H), 7.35 (t, $J = 8.0$ Hz, 1H), 7.24-7.22 (m, 1H), 7.05 (d, $J = 8.8$ Hz, 2H), 5.97 (s, 1H), 5.80 (s, 1H), 1.68 (s, 6H). All data are in accordance with the literature.²

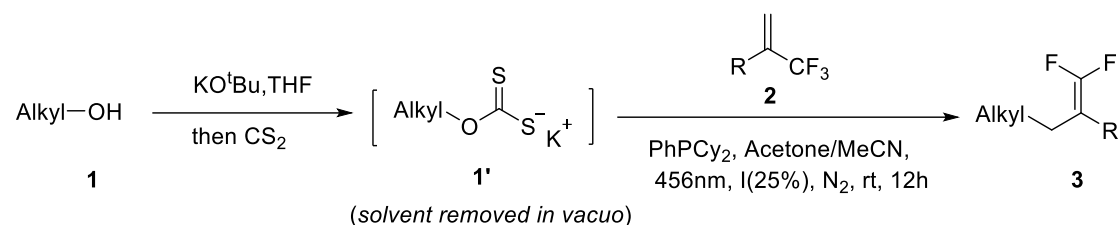


2-(3-Cyano-4-isobutoxyphenyl)-4-methyl-N-(3-(3,3,3-trifluoroprop-1-en-2-yl)phenyl)thiazole-5-carboxamide (2s) :

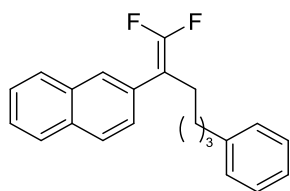
To a mixture of Febuxostat (0.95 g, 3.00 mmol, 1.0 equiv.) and oxalylchloride (0.51 mL, 6.00 mmol, 2.0 equiv.) in dry CH_2Cl_2 (12 mL) was added dropwise DMF (23.4 μL , 0.30 mmol, 0.1 equiv.). The reaction mixture was stirred at room temperature for 6 hours. Removal of the solvent in vacuo afforded the desired acid chloride which was used in the next step without further purification. To a mixture of 3-(3,3,3-trifluoroprop-1-en-2-yl)aniline (0.57 g, 3.00 mmol, 1.0 equiv.) and K_2CO_3 (0.42 g, 3.00 mmol, 1.0 equiv.) in dry THF (6 mL) was added dropwise a solution of the freshly prepared acid chloride (3.00 mmol, 1.0 equiv.) in dry THF (6 mL). This mixture was stirred at room temperature for 6 hours before water was added to quench the reaction. The resultant mixture was extracted with EtOAc (3*20 mL). The combined organic phases were dried over anhydrous Na_2SO_4 , filtered, and concentrated under reduced pressure. The resultant crude product was purified by column chromatography on silica gel (PE: EA=5:1) to give the desired trifluoromethyl alkene as a white solid 1.09 g (75% yield). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ : 8.16 (d, $J = 2.2$ Hz, 1H), 8.08 (dd, $J = 8.9$ Hz, $J = 2.3$ Hz, 1H), 7.67-7.62 (m, 2H), 7.54 (s, 1H), 7.40 (t, $J = 7.9$ Hz, 1H), 7.02 (d, $J = 8.9$ Hz, 1H), 6.01-6.00 (m, 1H), 5.84-5.82 (m, 1H), 3.91 (d, $J = 6.5$ Hz, 2H), 2.80 (s, 3H), 2.24-2.17 (m, 1H), 1.09 (d, $J = 6.7$ Hz, 6H). All data are in accordance with the literature.²

5. General Procedure for Deoxygenative Difluoroallylation of Xanthate Salts

Method C:



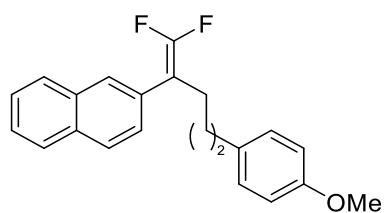
In a N₂-filled glovebox, an oven-dried 20 mL glass vial equipped with a magnetic stir bar was charged sequentially with alcohol **1** (1.0 eq), **KO^tBu** (1.1 eq), and **dry THF** (0.1 M). After being sealed with a septum cap and transferred out of the glovebox, the reaction mixture was stirred at room temperature for 30 minutes, followed by the addition of **CS₂** (3.0 eq) via microsyringe at 0°C and continued to be stirred for 3 hours at 0°C before removing the solvent in vacuo. The system was transferred into the glovebox, then **2** (1.2 eq), **PhPCy₂** (1.2 eq), and the mixed solvents of **Acetone/MeCN** (9/1, 0.05 M) were added. The vial was sealed with a septum cap and transferred out of the glovebox. The reaction mixture was stirred at room temperature under blue LEDs (456 nm, the light intensity is 25%) irradiation for 12 h. The solvent was removed in vacuo and the residue was purified by column chromatography to afford the product and chemoselectivity were determined from analysis of the reaction mixture by either ¹⁹F NMR.



2-(1,1-difluoro-6-phenylhex-1-en-2-yl)naphthalene (**3a**) :

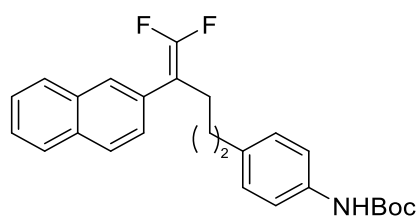
According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL, 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 83 mg (86% yield) of **3a** as a yellow oil: ¹H NMR (400 MHz, CDCl₃) δ: 7.87-7.79 (m, 4H), 7.54-7.45 (m, 3H), 7.29 (t, *J* = 7.2

Hz, 2H), 7.20 (dd, $J = 7.7$ Hz, 7.0 Hz, 3H), 2.63-2.56 (m, 4H), 1.74-1.66 (m, 2H), 1.53-1.46 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 153.8 (dd, $J = 288.9$ Hz, 285.1 Hz), 142.3, 133.2, 132.4, 131.1 (t, $J = 3.8$ Hz), 128.3, 128.2, 128.0, 127.9, 127.6, 127.3 (t, $J = 3.8$ Hz), 126.2, 126.1 (t, $J = 3.8$ Hz), 126.0, 125.7, 92.4 (dd, $J = 21.6$ Hz, 12.9 Hz), 35.5, 30.7, 27.4, 27.2; ^{19}F NMR (376 MHz, CDCl_3) δ : -91.01 (d, $J = 43.4$ Hz), -91.40 (d, $J = 43.3$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{22}\text{H}_{20}\text{F}_2$ ($\text{M}+\text{Na}$) $^+$ 345.1431, found 345.1434.



2-(1,1-difluoro-5-(4-methoxyphenyl)pent-1-en-2-yl)naphthalene (3b) : According to Method C, the reaction was carried out with alcohol **1b** (45.7 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF**

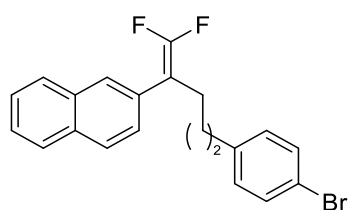
(3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 71 mg (70% yield) of **3b** as a colorless oil: ^1H NMR (400 MHz, CDCl_3) δ : 7.85-7.81 (m, 3H), 7.74 (s, 1H), 7.52-7.43 (m, 3H), 7.06 (d, $J = 8.7$ Hz, 2H), 6.83 (d, $J = 8.7$ Hz, 2H), 3.79 (s, 3H), 2.62-2.53 (m, 4H), 1.76-1.68 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 167.7, 153.8 (dd, $J = 289.4$ Hz, 285.6 Hz), 133.8, 133.2, 132.4, 131.0 (t, $J = 3.4$ Hz), 129.2, 128.0, 127.9, 127.6, 127.2 (t, $J = 3.4$ Hz), 126.2, 126.1, 126.1, 113.7, 92.3 (dd, $J = 21.6$ Hz, 12.9 Hz), 55.2, 34.2, 29.6, 27.1; ^{19}F NMR (376 MHz, CDCl_3) δ : -90.86 (d, $J = 41.9$ Hz), -91.19 (d, $J = 41.9$ Hz). All data are in accordance with the literature.²



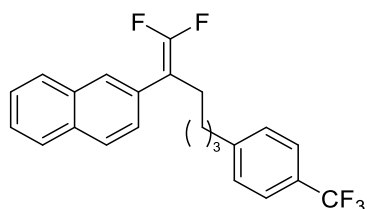
Tert-butyl (4-(5,5-difluoro-4-(naphthalen-2-yl)pent-4-en-1-yl)phenyl)carbamate (3c) : According to Method C, the reaction was carried out with alcohol **1c** (71.2 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂**

(54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 20:1) to afford

108 mg (82% yield) of **3c** as a white solid: $^1\text{H NMR}$ (400 MHz, CDCl_3) δ : 7.84-7.72 (m, 4H), 7.52-7.47 (m, 2H), 7.43 (d, $J = 8.6$ Hz, 1H), 7.25 (d, $J = 8.4$ Hz, 2H), 7.05 (d, $J = 8.6$ Hz, 2H), 6.45 (s, 1H), 2.61-2.51 (m, 4H), 1.74-1.66 (m, 2H), 1.53 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ : 153.7 (dd, $J = 288.9$ Hz, 285.6 Hz), 152.8, 136.4, 136.1, 133.2, 132.4, 130.9 (t, $J = 3.4$ Hz), 128.8, 128.0, 127.9, 127.5, 127.2 (t, $J = 3.4$ Hz), 126.2, 126.1, 118.6, 92.3 (dd, $J = 21.6$ Hz, 13.0 Hz), 80.3, 34.4, 29.4, 28.3, 27.1; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ : -90.85 (d, $J = 43.3$ Hz), -91.1 (d, $J = 43.3$ Hz); **HRMS** m/z (ESI) calcd for $\text{C}_{26}\text{H}_{27}\text{F}_2\text{NO}_2$ ($\text{M}+\text{Na}$) $^+$ 446.1908, found 446.1909.

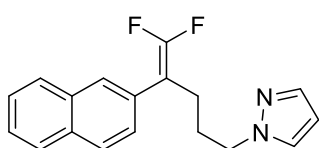


(5-(4-bromophenyl)-1,1-difluoropent-1-en-2-yl)naphthalene (3d) : According to Method C, the reaction was carried out with alcohol **1d** (60.3 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 83 mg (72% yield) of **3d** as a white solid: $^1\text{H NMR}$ (400 MHz, CDCl_3) δ : 7.85-7.80 (m, 3H), 7.71 (s, 1H), 7.52-7.49 (m, 2H), 7.41 (dd, $J = 8.6$ Hz, 8.5 Hz, 3H), 6.99 (d, $J = 8.4$ Hz, 2H), 2.61-2.52 (m, 4H), 1.75-1.67 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ : 153.8 (dd, $J = 288.9$ Hz, 285.6 Hz), 140.7, 133.2, 132.4, 131.3, 130.8 (t, $J = 3.8$ Hz), 130.1, 128.1, 127.9, 127.6, 127.2 (d, $J = 3.4$ Hz), 126.3, 126.2, 126.0 (d, $J = 2.9$ Hz), 119.5, 92.2 (dd, $J = 21.6$ Hz, 13.4 Hz), 34.5, 29.1, 27.1; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ : -90.75 (d, $J = 41.9$ Hz), -91.02 (d, $J = 43.4$ Hz). All data are in accordance with the literature.²



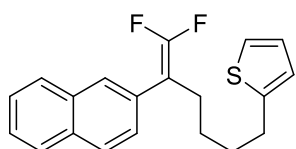
2-(1,1-difluoro-6-(4-(trifluoromethyl)phenyl)hex-1-en-2-yl)naphthalene (3e) : According to Method C, the reaction was carried out with alcohol **1e** (38.4 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 83 mg (71% yield) of **3e** as a colorless oil:

¹H NMR (400 MHz, CDCl₃) δ: 7.86-7.75 (m, 4H), 7.53-7.41 (m, 5H), 7.22 (d, *J* = 8.2 Hz, 2H), 2.64-2.53 (m, 4H), 1.70-1.63 (m, 2H), 1.49-1.41 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ: 153.8 (dd, *J* = 288.9 Hz, 285.6 Hz), 146.4, 133.2, 132.4, 130.9, 128.6, 127.9 (dt, *J* = 32.1 Hz, 25.4 Hz), 127.3 (d, *J* = 3.4 Hz), 126.3, 126.2, 126.1 (t, *J* = 3.4 Hz), 125.7 (d, *J* = 270.7 Hz), 125.2 (q, *J* = 3.4 Hz), 92.2 (dd, *J* = 21.1 Hz, 13.9 Hz), 35.3, 30.3, 27.3, 27.1; **¹⁹F NMR (376 MHz, CDCl₃)** δ: -62.13, -91.11 (d, *J* = 43.3 Hz), -91.30 (d, *J* = 43.3 Hz); **HRMS m/z (ESI)** calcd for C₂₃H₁₉F₅ (M+H)⁺ 391.1485, found 391.1481.



1-(5,5-difluoro-4-(naphthalen-2-yl)pent-4-en-1-yl)-1H-pyrazole (3f)

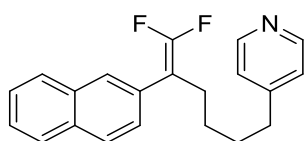
: According to Method C, the reaction was carried out with alcohol **1f** (33.6 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL, 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 10:1) to afford 31 mg (35% yield) of **3f** as a yellow oil: **¹H NMR (400 MHz, CDCl₃)** δ: 7.84-7.79 (m, 3H), 7.71 (s, 1H), 7.51-7.48 (m, 3H), 7.41 (m, 1H), 7.30 (d, *J* = 2.2 Hz, 1H), 4.13 (t, *J* = 7.0 Hz, 2H), 2.54-2.49 (m, 2H), 2.03-1.95 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ: 153.8 (dd, *J* = 289.4 Hz, 286.1 Hz), 139.3, 133.2, 132.5, 130.4 (t, *J* = 3.4 Hz), 129.0, 128.2, 127.9, 127.6, 127.2 (t, *J* = 3.8 Hz), 126.4, 126.2, 125.9 (t, *J* = 3.4 Hz), 105.3, 91.5 (dd, *J* = 21.6 Hz, 13.4 Hz), 51.1, 28.5, 24.7; **¹⁹F NMR (376 MHz, CDCl₃)** δ: -90.03 (d, *J* = 41.9 Hz), -90.34 (d, *J* = 41.9 Hz); **HRMS m/z (ESI)** calcd for C₁₈H₁₆F₂N₂ (M+H)⁺ 299.1360, found 299.1359.



2-(6,6-difluoro-5-(naphthalen-2-yl)hex-5-en-1-yl)thiophene (3g)

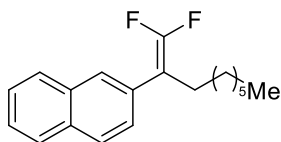
: According to Method C, the reaction was carried out with alcohol **1g** (38.5 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL, 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 10:1) to afford 41 mg (41% yield) of **3g** as a

colorless oil: $^1\text{H NMR}$ (400 MHz, CDCl_3) δ : 7.85-7.76 (m, 4H), 7.50 (td, $J = 3.6$ Hz, 8.7 Hz, 3H), 7.10 (d, $J = 5.2$ Hz, 1H), 6.89 (t, $J = 5.2$ Hz, 1H), 6.74 (d, $J = 3.7$ Hz, 1H), 2.80 (t, $J = 7.6$ Hz, 2H), 2.57-2.53 (m, 2H), 1.76-1.69 (m, 2H), 1.53-1.45 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ : 153.8 (dd, $J = 288.9$ Hz, 285.6 Hz), 145.2, 133.2, 132.4, 131.0(t, $J = 3.8$ Hz), 128.0, 127.9, 127.6, 127.3 (t, $J = 3.4$ Hz), 126.6, 126.2, 126.1, 124.0, 122.9, 92.3 (dd, $J = 21.6$ Hz, 13.4 Hz), 31.0, 29.5, 27.3, 27.1; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ : -91.05 (d, $J = 43.3$ Hz), -91.32 (d, $J = 43.3$ Hz); **HRMS** m/z (ESI) calcd for $\text{C}_{20}\text{H}_{18}\text{F}_2\text{S}$ ($\text{M}+\text{H}$) $^+$ 329.1176, found 329.1174.



4-(6,6-difluoro-5-(naphthalen-2-yl)hex-5-en-1-yl)pyridine (3h)

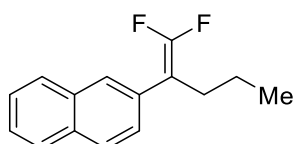
: According to Method C, the reaction was carried out with alcohol **1h** (41.2 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 5:1) to afford 50 mg (52% yield) of **3h** as a yellow oil: $^1\text{H NMR}$ (400 MHz, CDCl_3) δ : 7.44 (d, $J = 6.2$ Hz, 2H), 7.84-7.74 (m, 4H), 7.51-7.40 (m, 3H), 7.03 (d, $J = 6.3$ Hz, 2H), 2.57-2.51 (m, 4H), 1.69-1.61 (m, 2H), 1.48-1.40 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ : 153.8 (dd, $J = 288.9$ Hz, 285.6 Hz), 151.1, 149.6, 133.2, 132.4, 130.8, 128.1, 127.8, 127.6, 127.3 (t, $J = 3.4$ Hz), 126.3, 126.2, 126.0 (t, $J = 2.9$ Hz), 123.8, 92.1 (dd, $J = 21.1$ Hz, 13.4 Hz), 34.7, 29.4, 27.3, 27.0; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ : -91.04 (d, $J = 43.3$ Hz), -91.23 (d, $J = 43.3$ Hz); **HRMS** m/z (ESI) calcd for $\text{C}_{21}\text{H}_{19}\text{F}_2\text{N}$ ($\text{M}+\text{H}$) $^+$ 324.1564, found 324.1562.



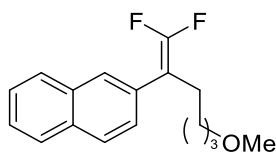
2-(1,1-difluoronon-1-en-2-yl)naphthalene (3i)

: According to Method C, the reaction was carried out with alcohol **1i** (30.7 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 71 mg (80% yield) of **3i** as a colorless oil: $^1\text{H NMR}$ (400 MHz, CDCl_3) δ : 7.86-7.79 (m, 4H), 7.51-7.45 (m, 3H), 2.54-2.49 (m, 2H), 1.43-1.25 (m, 10H), 0.88 (t,

$J = 7.1$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 153.8 (dd, $J = 288.9$ Hz, 285.1 Hz), 133.3, 132.4, 131.3 (t, $J = 3.4$ Hz), 128.0, 127.9, 127.6, 127.3 (t, $J = 3.4$ Hz), 126.2, 126.0, 92.6 (dd, $J = 21.6$ Hz, 12.9 Hz), 31.8, 29.0, 28.9, 27.8, 27.7, 22.6, 14.0; ^{19}F NMR (376 MHz, CDCl_3) δ : -91.4 (d, $J = 44.8$ Hz), -91.71 (d, $J = 44.8$ Hz). All data are in accordance with the literature.²

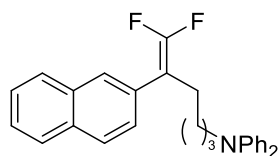


2-(1,1-difluoropent-1-en-2-yl)naphthalene (3j) : According to Method C, the reaction was carried out with alcohol **1j** (13.8 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 56 mg (80% yield) of **3j** as a colorless oil: ^1H NMR (400 MHz, CDCl_3) δ : 7.85-7.78 (m, 4H), 7.52-7.44 (m, 3H), 2.52-2.47 (m, 2H), 1.49-1.39 (m, 2H), 0.93 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 153.9 (dd, $J = 288.5$ Hz, 285.6 Hz), 133.2, 132.4, 131.3 (t, $J = 3.4$ Hz), 128.0, 127.9, 127.6, 127.3 (t, $J = 3.4$ Hz), 126.2, 126.0, 92.3 (dd, $J = 21.6$ Hz, 12.9 Hz), 29.6, 20.9, 13.4; ^{19}F NMR (376 MHz, CDCl_3) δ : -91.39 (d, $J = 44.8$ Hz), -91.61 (d, $J = 44.8$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{15}\text{H}_{14}\text{F}_2$ ($\text{M}+\text{H}$)⁺ 233.1142, found 233.1144.

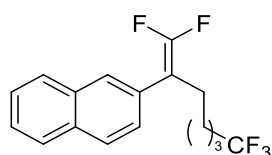


2-(1,1-difluoro-6-methoxyhex-1-en-2-yl)naphthalene (3k) : According to Method C, the reaction was carried out with alcohol **1k** (45.7 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:DCM = 10:1) to afford 58 mg (70% yield) of **3k** as a colorless oil: ^1H NMR (400 MHz, CDCl_3) δ : 7.85-7.79 (m, 4H), 7.51-7.44 (m, 3H), 3.34 (ts, $J = 6.6$ Hz, 5H), 2.58-2.53 (m, 2H), 1.66-1.59 (m, 2H), 1.52-1.45 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 153.8 (dd, $J = 289.4$ Hz, 285.1 Hz), 133.2, 132.4, 131.0 (t, $J = 3.4$ Hz), 128.0, 127.9, 127.6, 127.3 (t, $J = 3.4$ Hz), 126.2, 126.1 (t, $J = 3.4$ Hz), 126.0, 92.4 (dd, $J = 21.6$ Hz, 12.9 Hz), 72.4, 58.5, 28.9, 27.5, 24.4; ^{19}F NMR (376 MHz,

CDCl_3) δ : -91.08 (d, $J = 43.3$ Hz), -91.4 (d, $J = 43.3$ Hz); **HRMS m/z (ESI)** calcd for $\text{C}_{17}\text{H}_{18}\text{F}_2\text{O}$ ($\text{M}+\text{H}$)⁺ 277.1404, found 277.1406.

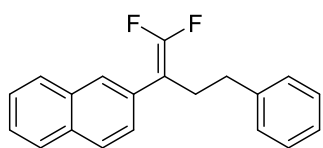


N-(6,6-difluoro-5-(naphthalen-2-yl)hex-5-en-1-yl)-N-phenylamine (3l) : According to Method C, the reaction was carried out with alcohol **1l** (68.1 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 20:1) to afford 53 mg (43% yield) of **3l** as a colorless oil: **¹H NMR (400 MHz, CDCl₃)** δ : 7.85-7.72 (m, 4H), 7.50 (t, $J = 4.8$ Hz, 2H), 7.39 (d, $J = 8.6$ Hz, 1H), 7.22 (t, $J = 7.4$ Hz, 4H), 6.96-6.88 (m, 6H), 3.63 (t, $J = 7.5$ Hz, 2H), 2.52 (t, $J = 7.4$ Hz, 2H), 1.73-1.65 (m, 2H), 1.47-1.40 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ : 153.8 (dd, $J = 288.9$ Hz, 285.1 Hz), 147.9, 133.2, 132.4, 130.9, 129.2, 128.1, 127.9, 127.6, 127.3 (t, $J = 3.4$ Hz), 126.3, 126.1, 121.1, 120.8, 92.3 (dd, $J = 21.1$ Hz, 13.4 Hz), 51.8, 27.4, 26.7, 25.1; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -91.10 (d, $J = 43.3$ Hz), -91.34 (d, $J = 43.3$ Hz); **HRMS m/z (ESI)** calcd for $\text{C}_{28}\text{H}_{25}\text{F}_2\text{N}$ ($\text{M}+\text{H}$)⁺ 414.2033, found 414.2034.



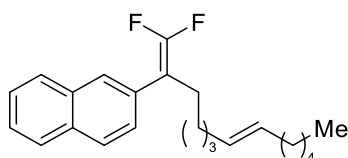
2-(1,1,7,7,7-pentafluorohept-1-en-2-yl)naphthalene (3m) : According to Method C, the reaction was carried out with alcohol **1m** (38.4 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 31 mg (33% yield) of **3m** as a colorless oil: **¹H NMR (400 MHz, CDCl₃)** δ : 7.86-7.76 (m, 4H), 7.53-7.41 (m, 3H), 2.57-2.52 (m, 2H), 2.06-2.00 (m, 2H), 1.64-1.56 (m, 2H), 1.51-1.43 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ : 153.9 (dd, $J = 288.0$ Hz, 285.6 Hz), 133.2, 132.5, 130.7, 128.4, 128.2, 127.9, 127.6, 127.3 (t, $J = 3.4$ Hz), 126.4, 126.2, 126.0 (t, $J = 3.4$ Hz), 125.7, 91.9 (dd, $J = 19.7$ Hz, 15.3 Hz), 33.5 (q, $J = 287.5$ Hz), 27.3, 26.7, 21.2, 21.2; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -66.30 (t, $J = 10.1$ Hz), -90.88 (dd, $J = 14.5$ Hz, 14.4 Hz);

HRMS m/z (ESI) calcd for $C_{17}H_{15}F_5$ ($M+H$)⁺ 315.1172, found 315.1173.



2-(1,1-difluoro-4-phenylbut-1-en-2-yl)naphthalene (3n) :

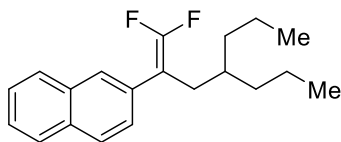
According to Method C, the reaction was carried out with alcohol **1n** (32.4 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μ L, 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 40 mg (36% yield) of **3n** as a white solid: **¹H NMR (400 MHz, CDCl₃)** δ : 7.89-7.79 (m, 4H), 7.55-7.46 (m, 3H), 7.31 (t, J = 7.6 Hz, 2H), 7.24-7.16 (m, 3H), 2.87-2.82 (m, 2H), 2.74-2.71 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ : 153.9 (dd, J = 289.9 Hz, 285.6 Hz), 133.3, 132.5, 130.8 (t, J = 3.8 Hz), 128.4, 128.3, 128.1, 127.9, 127.6, 127.4 (t, J = 3.8 Hz), 126.3, 126.2, 126.1, 92.0 (dd, J = 21.6 Hz, 13.4 Hz), 34.0, 29.7; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -90.42 (d, J = 41.9 Hz), -91.06 (d, J = 41.9 Hz); **HRMS m/z (ESI)** calcd for $C_{20}H_{16}F_2$ ($M+H$)⁺ 295.1298, found 295.1295.



(E)-2-(1,1-difluorotrideca-1,7-dien-2-yl)naphthalene

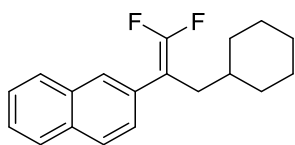
(3o) : According to Method C, the reaction was carried out with alcohol **1o** (46.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μ L, 0.9

mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 50 mg (49% yield) of **3o** as a colorless oil: **¹H NMR (400 MHz, CDCl₃)** δ : 7.84-7.76 (m, 4H), 7.50-7.42 (m, 3H), 5.36-5.29 (m, 2H), 2.53-2.49 (m, 2H), 2.02-1.95 (m, 4H), 1.43-1.24 (m, 10H), 0.88 (t, J = 7.1 Hz, 3H), **¹³C NMR (100 MHz, CDCl₃)** δ : 153.8 (dd, J = 288.5 Hz, 285.1 Hz), 133.3, 132.4, 131.2 (t, J = 3.8 Hz), 130.3, 129.3, 128.0, 127.9, 127.6, 127.3 (t, J = 3.4 Hz), 126.2, 126.2 (t, J = 3.4 Hz), 126.0, 92.5 (dd, J = 22.1 Hz, 12.5 Hz), 31.5, 29.4, 29.0, 27.6, 27.3, 27.1, 26.8, 22.5, 14.0; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -91.32 (d, J = 44.8 Hz), -91.61 (d, J = 44.8 Hz); **HRMS m/z (ESI)** calcd for $C_{23}H_{28}F_2$ ($M+H$)⁺ 343.2237, found 343.2238.



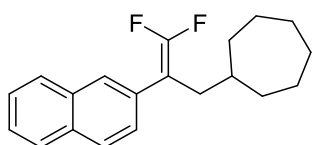
2-(1,1-difluoro-4-propylhept-1-en-2-yl)naphthalene

(3p) : According to Method C, the reaction was carried out with alcohol **1p** (34.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL, 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 72 mg (78% yield) of **3p** as a colorless oil: ¹H NMR (400 MHz, CDCl₃) δ: 7.85-7.82 (m, 3H), 7.77 (s, 1H), 7.52-7.43 (m, 3H), 2.45-2.42 (m, 2H), 1.36-1.21 (m, 9H), 0.87-0.80 (m, 6H); ¹³C NMR (100 MHz, CDCl₃) δ: 154.1 (dd, *J* = 287.5 Hz, 286.1 Hz), 133.2, 132.4, 131.4, 127.9, 127.8, 127.6, 127.4 (t, *J* = 3.4 Hz), 126.2 (t, *J* = 2.9 Hz), 126.1, 126.0, 91.8 (dd, *J* = 19.6 Hz, 14.4 Hz), 35.3, 35.0, 32.1, 19.4, 14.4; ¹⁹F NMR (376 MHz, CDCl₃) δ: -91.28, -91.30; HRMS *m/z* (ESI) calcd for C₂₀H₂₄F₂ (M+H)⁺ 303.1924, found 303.1923.



2-(3-cyclohexyl-1,1-difluoroprop-1-en-2-yl)naphthalene

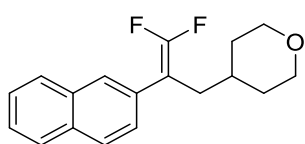
(3q) : According to Method C, the reaction was carried out with alcohol **1q** (30.0 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL, 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 49 mg (57% yield) of **3q** as a white solid: ¹H NMR (400 MHz, CDCl₃) δ: 7.84-7.77 (m, 4H), 7.51-7.43 (m, 3H), 2.40-2.37 (m, 2H), 1.74-1.58 (m, 5H), 1.30-1.25 (m, 1H), 1.15-1.04 (m, 3H), 0.99-0.91 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 154.1 (dd, *J* = 288.9 Hz, 285.1 Hz), 133.2, 132.4, 131.4, 127.9, 127.9, 127.6, 127.3 (t, *J* = 3.4 Hz), 126.2 (t, *J* = 3.4 Hz), 126.2, 126.0, 91.2 (dd, *J* = 22.5 Hz, 12.5 Hz), 35.7, 35.3, 32.9, 26.4, 26.0; ¹⁹F NMR (376 MHz, CDCl₃) δ: -90.82 (d, *J* = 43.3 Hz), -91.53 (d, *J* = 43.3 Hz). All data are in accordance with the literature.⁷



2-(3-cycloheptyl-1,1-difluoroprop-1-en-2-yl)naphthalene

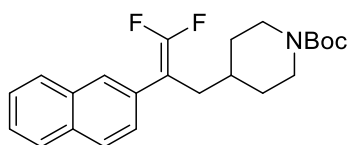
(3r) : According to Method C, the reaction was carried out with alcohol **1r** (34.3 mg, 0.3 mmol), **KO^tBu** (37.0 mg,

0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μ L, 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 64 mg (71% yield) of **3r** as a white solid: **¹H NMR (400 MHz, CDCl₃)** δ : 7.85-7.76 (m, 4H), 7.50-7.42 (dt, J = 3.7 Hz, 8.6 Hz, 3H), 2.42-2.39 (m, 2H), 1.72-1.68 (m, 2H), 1.64-1.57 (m, 2H), 1.53-1.42 (m, 5H), 1.34-1.17 (m, 4H); **¹³C NMR (100 MHz, CDCl₃)** δ : 154.3 (dd, J = 288.5 Hz, 284.6 Hz), 133.2, 132.4, 131.4 (t, J = 4.3 Hz), 128.0, 127.9, 127.6, 127.3 (t, J = 2.9 Hz), 126.3 (t, J = 3.4 Hz), 126.2, 126.0, 91.8 (dd, J = 21.6 Hz, 12.5 Hz), 37.1, 35.7, 34.0, 28.4, 26.0; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -91.13 (d, J = 43.2 Hz), -91.67 (d, J = 43.3 Hz). All data are in accordance with the literature.²



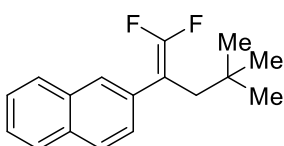
4-(3,3-difluoro-2-(naphthalen-2-yl)allyl)tetrahydro-2H-pyran (3s) : According to Method C, the reaction was carried out with alcohol **1s** (30.6 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μ L,

0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 20:1) to afford 44 mg (49% yield) of **3s** as a yellow oil: **¹H NMR (400 MHz, CDCl₃)** δ : 7.87-7.77 (m, 4H), 7.53-7.43 (m, 3H), 3.93-3.89 (m, 2H), 3.24 (t, J = 11.8 Hz, 2H), 2.48-2.45 (m, 2H), 1.66-1.48 (m, 3H), 1.39-1.32 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ : 154.2 (dd, J = 289.9 Hz, 285.1 Hz), 133.2, 132.4, 131.1 (t, J = 3.8 Hz), 128.1, 127.9, 127.6, 127.3 (t, J = 3.4 Hz), 126.3, 126.2, 126.0 (t, J = 2.9 Hz), 90.5 (dd, J = 22.0 Hz, 12.9 Hz), 67.7, 34.8, 33.2, 32.6; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -90.23 (d, J = 41.9 Hz), -90.81 (d, J = 41.9 Hz). All data are in accordance with the literature.⁷



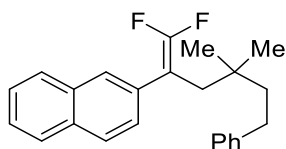
Tert-butyl 4-(3,3-difluoro-2-(naphthalen-2-yl) allyl) piperidine-1-carboxylate (3t) : According to Method C, the reaction was carried out with alcohol **1t** (60.3 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μ L, 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36

mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 10:1) to afford 50 mg (43% yield) of **3t** as a colorless oil: ¹H NMR (400 MHz, CDCl₃) δ: 7.85-7.76 (m, 4H), 7.51-7.42 (m, 3H), 4.20-3.87 (m, 2H), 2.54 (td, *J* = 12.5 Hz, 7.3 Hz, 4H), 1.68-1.59 (m, 3H), 1.44(s, 9H), 1.20-1.13 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 154.7, 154.3 (dd, *J* = 289.4 Hz, 285.6 Hz), 133.2, 132.4, 131.0 (t, *J* = 3.8 Hz), 128.2, 127.9, 127.6, 127.3 (t, *J* = 3.4 Hz), 126.3, 126.2, 126.0 (t, *J* = 3.4 Hz), 90.7 (dd, *J* = 22.0 Hz, 12.9 Hz), 79.3, 34.4, 34.2, 31.7, 28.4; ¹⁹F NMR (376 MHz, CDCl₃) δ: -90.17 (d, *J* = 41.9 Hz), -90.80 (d, *J* = 41.9 Hz). All data are in accordance with the literature.⁷



2-(1,1-difluoro-4,4-dimethylpent-1-en-2-yl)naphthalene

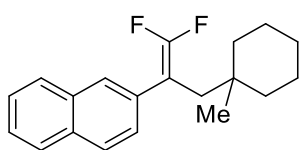
(3u) : According to Method C, the reaction was carried out with alcohol **1u** (22.2 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL, 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 47 mg (60% yield) of **3u** as a white solid: ¹H NMR (400 MHz, CDCl₃) δ: 7.84-7.79 (m, 4H), 7.50-7.44 (m, 3H), 2.46(m, 2H), 0.83 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ: 154.6 (dd, *J* = 288.9 Hz, 286.5 Hz), 133.2, 133.1 (t, *J* = 4.3 Hz), 132.3, 127.8, 127.6, 127.4 (t, *J* = 2.9 Hz), 126.5 (t, *J* = 2.9 Hz), 126.2, 125.9, 91.2 (dd, *J* = 21.6 Hz, 12.5 Hz), 41.2, 32.8, 29.7; ¹⁹F NMR (376 MHz, CDCl₃) δ: -89.16 (d, *J* = 40.4 Hz), -92.00 (d, *J* = 40.5 Hz). All data are in accordance with the literature.⁸



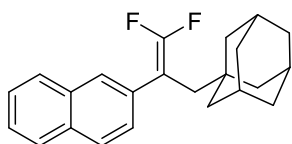
2-(1,1-difluoro-4,4-dimethyl-6-phenylhex-1-en-2-yl)naphthalene

(3v) : According to Method C, the reaction was carried out with alcohol **1v** (49.3 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL, 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 77 mg (73% yield) of **3v** as

a white solid: $^1\text{H NMR}$ (400 MHz, CDCl_3) δ : 7.86-7.82 (m, 4H), 7.52-7.47 (m, 3H), 7.17-7.09 (m, 3H), 6.88 (dd, $J = 2.1$ Hz, 1.7 Hz, 2H), 2.55 (t, $J = 2.5$ Hz, 2H), 2.50-2.45 (m, 2H), 1.50-1.46 (m, 2H), 0.89 (s, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ : 154.6 (dd, $J = 288.5$ Hz, 287.0 Hz), 142.9, 133.3, 133.0, 132.4, 128.2, 128.0, 127.8, 127.6, 127.5 (t, $J = 2.9$ Hz), 126.5 (t, $J = 2.4$ Hz), 126.3, 126.0, 125.5, 90.1 (dd, $J = 21.6$ Hz, 13.4 Hz), 44.5, 39.1, 35.5, 30.6, 27.4; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ : -88.83 (d, $J = 40.4$ Hz), -91.45 (d, $J = 39.0$ Hz), **HRMS m/z (ESI)** calcd for $\text{C}_{24}\text{H}_{24}\text{F}_2$ ($\text{M}+\text{H}$) $^+$ 351.1924, found 351.1925.

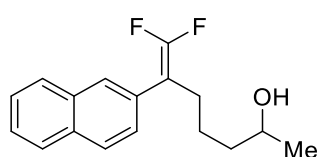


2-(1,1-difluoro-3-(1-methylcyclohexyl)prop-1-en-2-yl)naphthalene (3w) : According to Method C, the reaction was carried out with alcohol **1w** (34.3 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 76 mg (84% yield) of **3w** as a white solid: $^1\text{H NMR}$ (400 MHz, CDCl_3) δ : 7.85-7.79 (m, 4H), 7.52-7.46 (m, 3H), 2.48 (t, $J = 2.6$ Hz, 2H), 1.43-1.29 (m, 5H), 1.24-1.16 (m, 5H), 0.80 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ : 154.6 (dd, $J = 288.5$ Hz, 286.5 Hz), 133.3, 132.3, 127.9, 127.8, 127.6, 127.4 (t, $J = 2.9$ Hz), 126.6 (t, $J = 2.4$ Hz), 126.1, 125.9, 90.7 (dd, $J = 21.6$ Hz, 12.9 Hz), 40.4, 38.0, 35.2, 26.2, 24.6, 22.0; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ : -88.81 (d, $J = 40.4$ Hz), -91.76 (d, $J = 40.4$ Hz). All data are in accordance with the literature.⁹



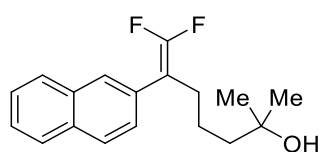
(3r,5r,7r)-1-(3,3-difluoro-2-(naphthalen-2-yl)allyl)adamantane (3x) : According to Method C, the reaction was carried out with alcohol **1x** (45.7 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 80 mg (79% yield) of **3x** as a white solid: $^1\text{H NMR}$ (400 MHz, CDCl_3) δ : 7.84-7.79 (m, 4H), 7.51-7.45 (m,

3H), 2.33 (t, $J = 2.7$ Hz, 2H), 1.89-1.82 (m, 3H), 1.64-1.51 (m, 6H), 1.44-1.38 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ : 154.6 (dd, $J = 289.4$ Hz, 286.1 Hz), 133.4, 133.2, 132.3, 127.9, 127.8, 127.6, 127.2 (t, $J = 3.4$ Hz), 126.5 (t, $J = 2.9$ Hz), 126.1, 125.9, 89.9 (dd, $J = 21.6$ Hz, 12.5 Hz), 42.6, 42.0, 36.8, 34.7, 28.5; ^{19}F NMR (376 MHz, CDCl_3) δ : -88.37 (d, $J = 39.0$ Hz), -91.70 (d, $J = 40.5$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{23}\text{H}_{24}\text{F}_2$ ($\text{M}+\text{H}$) $^+$ 339.1924, found 339.1921.



7,7-difluoro-6-(naphthalen-2-yl)hept-6-en-2-ol (3y) :

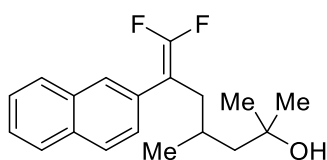
According to Method C, the reaction was carried out with alcohol **1y** (27.0 mg, 0.3 mmol), KO^tBu (37.0 mg, 0.33 mmol), dry THF (3.0 mL), CS_2 (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), PhPCy_2 (98.8 mg, 0.36 mmol), Acetone/MeCN (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 5:1) to afford 37 mg (45% yield) of **3y** as a yellow oil: ^1H NMR (400 MHz, CDCl_3) δ : 7.85-7.78 (m, 4H), 7.51-7.43 (m, 3H), 3.73-3.72 (m, 1H), 2.56-2.51 (m, 2H), 1.52-1.41 (m, 4H), 1.51 (d, $J = 6.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 153.8 (dd, $J = 288.9$ Hz, 285.6 Hz), 133.2, 132.4, 131.0 (t, $J = 3.4$ Hz), 128.0, 127.9, 127.6, 127.3 (t, $J = 3.4$ Hz), 126.3, 126.1, 92.3 (dd, $J = 21.6$ Hz, 12.9 Hz), 67.8, 38.4, 27.6, 23.9, 23.5; ^{19}F NMR (376 MHz, CDCl_3) δ : -91.06 (d, $J = 43.4$ Hz), -91.36 (d, $J = 43.3$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{17}\text{H}_{18}\text{F}_2\text{O}$ ($\text{M}+\text{H}$) $^+$ 277.1404, found 277.1405.



7,7-difluoro-2-methyl-6-(naphthalen-2-yl)hept-6-en-2-ol (3z) :

According to Method C, the reaction was carried out with alcohol **1z** (31.2 mg, 0.3 mmol), KO^tBu (37.0 mg, 0.33 mmol), dry THF (3.0 mL), CS_2 (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), PhPCy_2 (98.8 mg, 0.36 mmol), Acetone/MeCN (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 5:1) to afford 58 mg (67% yield) of **3z** as a yellow solid: ^1H NMR (400 MHz, CDCl_3) δ : 7.84-7.77 (m, 4H), 7.51-7.43 (m, 3H), 2.54-2.50 (m, 2H), 1.54-1.43 (m, 4H), 1.14 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ : 153.8 (dd, $J = 289.4$ Hz, 285.1 Hz), 133.2, 132.4, 131.0 (t, $J = 3.8$ Hz),

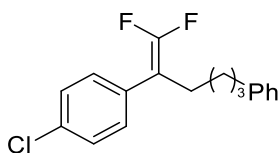
128.1, 127.9, 127.6, 127.3 (t, $J = 3.4$ Hz), 126.2, 126.1 (t, $J = 3.4$ Hz), 126.0, 92.4 (dd, $J = 21.6$ Hz, 12.9 Hz), 70.8, 43.0, 29.2, 28.1, 22.1; ^{19}F NMR (376 MHz, CDCl_3) δ : -91.06 (d, $J = 43.3$ Hz), -91.40 (d, $J = 43.4$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{18}\text{H}_{20}\text{F}_2\text{O}$ ($\text{M}+\text{Na}$) $^+$ 313.1380, found 313.1384.



7,7-difluoro-2,4-dimethyl-6-(naphthalen-2-yl)hept-6-en

-2-ol (3aa) : According to Method C, the reaction was carried out with alcohol **1aa** (35.5 mg, 0.3 mmol), KO^tBu (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), CS_2 (54.1 μL ,

0.9 mmol), and then trifluoromethyl alkene **2a** (80.0 mg, 0.36 mmol), PhPCy_2 (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 5:1) to afford 47 mg (52% yield) of **3aa** as a yellow oil: ^1H NMR (400 MHz, CDCl_3) δ : 7.85-7.80 (m, 4H), 7.52-7.46 (m, 3H), 2.70-2.64 (m, 1H), 2.37-2.31 (m, 1H), 1.70-1.54 (m, 2H), 1.40-1.34 (m, 1H), 1.14 (d, $J = 4.9$ Hz, 6H), 0.99 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 154.4 (dd, $J = 289.4$ Hz, 284.6 Hz), 133.3, 133.2, 133.0, 132.7, 132.4, 131.1 (t, $J = 3.8$ Hz), 129.0, 128.7, 128.4, 128.0, 127.9, 127.6, 127.4 (t, $J = 3.4$ Hz), 126.5, 126.3, 126.2, 126.1 (t, $J = 3.4$ Hz), 126.0, 91.7 (dd, $J = 21.6$ Hz, 12.5 Hz), 71.4, 71.2, 51.0, 49.8, 49.3, 37.9, 36.2, 30.2, 30.0, 29.9, 29.8, 27.7, 26.6, 25.8, 23.2, 21.4, 20.6; ^{19}F NMR (376 MHz, CDCl_3) δ : -69.14 (d, $J = 10.1$ Hz), -90.59 (d, $J = 41.9$ Hz), -91.35 (d, $J = 41.9$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{19}\text{H}_{22}\text{F}_2\text{O}$ ($\text{M}+\text{Na}$) $^+$ 327.1536, found 327.1538.

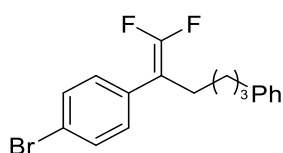


1-chloro-4-(1,1-difluoro-6-phenylhex-1-en-2-yl)benzene

(3ab) : According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), KO^tBu (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), CS_2 (54.1 μL , 0.9 mmol), and

then trifluoromethyl alkene **2b** (74.2 mg, 0.36 mmol), PhPCy_2 (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 42 mg (46% yield) of **3ab** as a colorless oil: ^1H NMR (400 MHz, CDCl_3) δ : 7.34-7.26 (m, 4H), 7.23-7.13 (m, 5H), 2.58 (t, $J = 7.8$ Hz, 2H), 2.44-2.39 (m, 2H), 1.67-1.59 (m, 2H), 1.44-1.37 (m, 2H); ^{13}C NMR (100

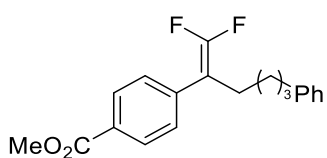
MHz, CDCl₃) δ : 153.6 (dd, $J = 288.5$ Hz, 286.1 Hz), 142.2, 133.0, 132.1, 129.5 (t, $J = 3.4$ Hz), 128.6, 128.3, 128.2, 125.7, 91.5 (dd, $J = 20.1$ Hz, 14.9 Hz), 35.5, 30.6, 27.2, 27.1; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -90.82, -90.84. All data are in accordance with the literature.¹⁰



1-bromo-4-(1,1-difluoro-6-phenylhex-1-en-2-yl)benzene

(3ac) : According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μ L, 0.9 mmol),

and then trifluoromethyl alkene **2c** (90.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 52 mg (50% yield) of **3ac** as a colorless oil: **¹H NMR (400 MHz, CDCl₃)** δ : 7.49 (d, $J = 8.6$ Hz, 2H), 7.27 (t, $J = 7.2$ Hz, 2H), 7.20-7.12 (m, 5H), 2.58 (t, $J = 7.5$ Hz, 2H), 2.43-2.38 (m, 2H), 1.66-1.58 (m, 2H), 1.43-1.36 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ : 153.5 (dd, $J = 288.5$ Hz, 286.5 Hz), 142.2, 132.6, 131.6, 129.9 (t, $J = 3.4$ Hz), 128.3, 128.2, 125.7, 121.1, 91.5 (dd, $J = 21.6$ Hz, 13.9 Hz), 35.4, 30.6, 27.2, 27.1; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -90.67 (d, $J = 43.3$ Hz), -90.83 (d, $J = 43.3$ Hz); **HRMS m/z (ESI)** calcd for C₁₈H₁₇BrF₂ (M+Na)⁺ 373.0379, found 373.0381.

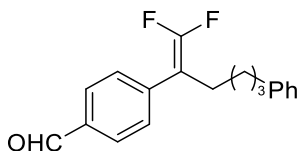


Methyl 4-(1,1-difluoro-6-phenylhex-1-en-2-yl) benzoate

(3ad): According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μ L, 0.9 mmol),

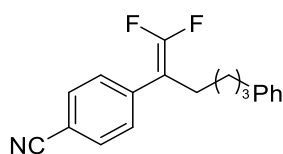
and then trifluoromethyl alkene **2d** (82.8 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 20:1) to afford 79 mg (80% yield) of **3ad** as a colorless oil: **¹H NMR (400 MHz, CDCl₃)** δ : 8.04 (d, $J = 8.6$ Hz, 2H), 7.38 (d, $J = 8.7$ Hz, 2H), 7.29-7.25 (m, 2H), 7.18 (td, $J = 7.2$ Hz, 6.8 Hz, 3H), 3.93 (s, 3H), 2.58 (t, $J = 7.9$ Hz, 2H), 2.49-2.44 (m, 2H), 1.67-1.60 (m, 2H), 1.45-1.38 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ : 166.7, 153.8 (dd, $J = 290.9$ Hz, 286.6 Hz), 142.2, 138.5 (t, $J = 3.8$ Hz), 129.7, 128.8, 128.3, 128.2, 128.1 (t, $J = 3.4$ Hz), 125.7, 92.0 (dd, $J =$

22.0 Hz, 12.5 Hz), 52.1, 35.4, 30.6, 27.2, 27.0; ^{19}F NMR (376 MHz, CDCl_3) δ : -89.28 (d, $J = 39.0$ Hz), -89.52 (d, $J = 39.0$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{20}\text{H}_{20}\text{F}_2$ ($\text{M}+\text{H}$) $^+$ 331.1510, found 331.1509.



4-(1,1-difluoro-6-phenylhex-1-en-2-yl)benzaldehyde

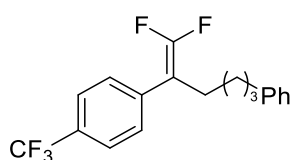
(3ae) : According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2e** (72.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 10:1) to afford 43 mg (43% yield) of **3ae** as a yellow oil: ^1H NMR (400 MHz, CDCl_3) δ : 10.01 (s, 1H), 7.87-7.85 (d, $J = 8.3$ Hz, 2H), 7.47 (d, $J = 6.9$ Hz, 2H), 7.28-7.24 (m, 2H), 7.19-7.12 (m, 3H), 2.58 (t, $J = 7.8$ Hz, 2H), 2.50-2.45 (m, 2H), 1.68-1.60 (m, 2H), 1.46-1.38 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 191.6, 153.9 (dd, $J = 291.3$ Hz, 287.5 Hz), 142.1, 140.2 (t, $J = 4.3$ Hz), 135.1, 129.8, 128.7 (t, $J = 3.4$ Hz), 128.3, 125.8, 92.1 (dd, $J = 22.5$ Hz, 11.5 Hz), 35.4, 30.6, 27.2, 27.0; ^{19}F NMR (376 MHz, CDCl_3) δ : -88.36 (d, $J = 37.6$ Hz), -88.74 (d, $J = 36.1$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{19}\text{H}_{18}\text{F}_2\text{O}$ ($\text{M}+\text{H}$) $^+$ 301.1404, found 301.1407.



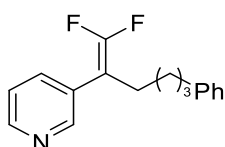
4-(1,1-difluoro-6-phenylhex-1-en-2-yl)benzotrile (3af) :

According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2f** (71.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 20:1) to afford 44 mg (49% yield) of **3af** as a colorless oil: ^1H NMR (400 MHz, CDCl_3) δ : 7.64 (d, $J = 8.4$ Hz, 2H), 7.39 (d, $J = 8.2$ Hz, 2H), 7.08-7.25 (m, 2H), 7.18 (t, $J = 7.1$ Hz, 1H), 7.13 (d, $J = 6.8$ Hz, 2H), 2.58 (t, $J = 7.8$ Hz, 2H), 2.47-2.42 (m, 2H), 1.66-1.59 (m, 2H), 1.43-1.36 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 153.9 (dd, $J = 291.3$ Hz, 287.8 Hz), 142.0, 138.7, 132.2, 128.8 (t, $J = 3.4$ Hz), 128.3, 125.8, 118.7, 110.9, 91.7 (dd, $J = 23.0$ Hz, 11.5 Hz), 35.4,

30.5, 27.1, 26.9; ^{19}F NMR (376 MHz, CDCl_3) δ : -87.94 (d, $J = 36.1$ Hz), -88.49 (d, $J = 36.1$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{19}\text{H}_{17}\text{F}_2\text{N}$ ($\text{M}+\text{H}$) $^+$ 298.1407, found 298.1408.

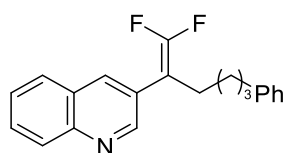


1-(1,1-difluoro-6-phenylhex-1-en-2-yl)-4-(trifluoromethyl)benzene (3ag) : According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2g** (70.9 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 65 mg (64% yield) of **3ag** as a colorless oil: ^1H NMR (400 MHz, CDCl_3) δ : 7.62 (d, $J = 8.4$ Hz, 2H), 7.41 (d, $J = 8.3$ Hz, 2H), 7.32-7.25 (m, 3H), 7.20-7.12 (m, 3H), 2.58 (t, $J = 7.9$ Hz, 2H), 2.48-2.43 (m, 2H), 1.67-1.59 (m, 2H), 1.44-1.37 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 153.8 (t, $J = 287.9$ Hz), 142.1, 137.5, 129.4 (q, $J = 32.6$ Hz), 128.5, 128.3, 125.8, 125.4, 122.7, 91.7 (dd, $J = 22.0$ Hz, 12.5 Hz), 35.4, 30.6, 27.1; ^{19}F NMR (376 MHz, CDCl_3) δ : -62.51, -89.55 (d, $J = 40.5$ Hz), -89.95 (d, $J = 39.0$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{19}\text{H}_{17}\text{F}_5$ ($\text{M}+\text{H}$) $^+$ 341.1329, found 341.1330.



3-(1,1-difluoro-6-phenylhex-1-en-2-yl)pyridine (3ah) : According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL , 0.9 mmol), and then trifluoromethyl alkene **2h** (62.3 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 10:1) to afford 58 mg (71% yield) of **3ah** as a yellow oil: ^1H NMR (400 MHz, CDCl_3) δ : 8.56-8.51 (m, 2H), 7.59 (d, $J = 8.0$ Hz, 1H), 7.30-7.10 (m, 6H), 2.58 (t, $J = 7.8$ Hz, 2H), 2.46-2.41 (m, 2H), 1.67-1.59 (m, 2H), 1.45-1.38 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 153.8 (dd, $J = 289.9$ Hz, 287.0 Hz), 149.2 (t, $J = 3.8$ Hz), 148.3, 142.1, 135.5, 129.7, 128.3, 125.7, 123.3, 89.7 (dd, $J = 24.0$ Hz, 12.5 Hz), 35.4, 30.6, 27.1, 27.0; ^{19}F NMR (376 MHz, CDCl_3) δ : -89.42 (d, $J = 40.4$ Hz), -90.14 (d, $J = 40.5$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{17}\text{H}_{17}\text{F}_2\text{N}$ ($\text{M}+\text{H}$) $^+$ 274.1407,

found 274.1409.



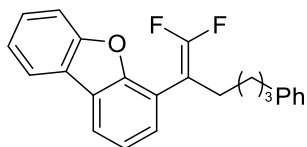
3-(1,1-difluoro-6-phenylhex-1-en-2-yl)quinoline (3ai) :

According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μ L, 0.9 mmol), and then trifluoromethyl alkene **2i** (80.3 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 10:1) to afford 75 mg (76% yield) of **3ai** as a yellow oil: **¹H NMR (400 MHz, CDCl₃)** δ : 8.88 (t, J = 2.0 Hz, 1H), 8.13-8.03 (m, 2H), 7.81-7.71 (m, 2H), 7.58 (t, J = 8.2 Hz, 1H), 7.26-7.11 (m, 5H), 2.60-2.53 (m, 4H), 1.70-1.63 (m, 2H), 1.50-1.42 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ : 154.1 (dd, J = 290.4 Hz, 287.5 Hz), 150.1, 146.8, 142.1, 135.0, 129.7, 129.1, 128.3, 127.7, 127.6, 127.1, 126.9 (t, J = 4.3 Hz), 125.8, 89.9 (dd, J = 23.5 Hz, 12.9 Hz), 35.4, 30.6, 27.1; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -89.01 (d, J = 39.0 Hz), -89.85 (d, J = 40.4 Hz); **HRMS m/z (ESI)** calcd for C₂₁H₁₉F₂N (M+H)⁺ 324.1564, found 324.1567.



5-(1,1-difluoro-6-phenylhex-1-en-2-yl)pyrimidine (3aj) :

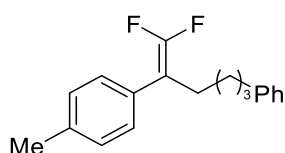
According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μ L, 0.9 mmol), and then trifluoromethyl alkene **2j** (62.7 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 20:1) to afford 38 mg (46% yield) of **3aj** as a yellow oil: **¹H NMR (400 MHz, CDCl₃)** δ : 9.12 (s, 1H), 8.68 (s, 1H), 7.27 (t, J = 7.6 Hz, 2H), 7.19-7.12 (m, 3H), 2.59 (t, J = 7.7 Hz, 2H), 2.47-2.43 (m, 2H), 1.68-1.60 (m, 2H), 1.47-1.39 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ : 157.1, 155.8 (t, J = 3.8 Hz), 154.2 (dd, J = 293.2 Hz, 288.9 Hz), 141.8, 128.3, 128.2, 128.0 (t, J = 4.3 Hz), 125.9, 87.2 (dd, J = 26.9 Hz, 12.5 Hz), 35.4, 30.5, 27.0, 26.3; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -86.79 (d, J = 36.1 Hz), -87.95 (d, J = 36.1 Hz); **HRMS m/z (ESI)** calcd for C₁₆H₁₆F₂N₂ (M+H)⁺ 275.1360, found 275.1363.



2-(1,1-difluoro-6-phenylhex-1-en-2-yl)dibenzo[b,d]furan

(3ak) : According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL, 0.9 mmol),

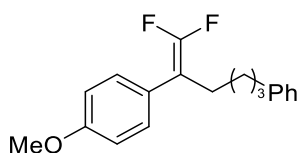
and then trifluoromethyl alkene **2k** (94.3 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 10:1) to afford 75 mg (71% yield) of **3ak** as a yellow oil: **¹H NMR (400 MHz, CDCl₃)** δ: 8.00 (d, *J* = 7.6 Hz, 1H), 7.94 (dd, *J* = 2.3 Hz, 2.4 Hz, 1H), 7.63 (d, *J* = 8.2 Hz, 1H), 7.53-7.48 (m, 1H), 7.41-7.35 (m, 3H), 7.23 (t, *J* = 7.6 Hz, 2H), 7.18-7.11 (m, 3H), 2.69-2.64 (m, 2H), 2.57 (t, *J* = 7.8 Hz, 2H), 1.73-1.65 (m, 2H), 1.47-1.39 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ: 156.0, 153.9, 153.5 (dd, *J* = 288.5 Hz, 286.1 Hz), 142.4, 128.3, 128.2, 127.8, 127.3, 125.6, 124.5, 124.1, 122.8, 122.7, 120.7, 120.0, 118.3 (d, *J* = 5.3 Hz), 111.8, 88.1 (dd, *J* = 24.9 Hz, 14.9 Hz), 35.5, 30.6, 27.3, 27.2; **¹⁹F NMR (376 MHz, CDCl₃)** δ: -88.12 (d, *J* = 40.4 Hz), -91.63 (d, *J* = 40.4 Hz); **HRMS m/z (ESI)** calcd for C₂₄H₂₀F₂O (M+Na)⁺ 385.1380, found 385.1382.



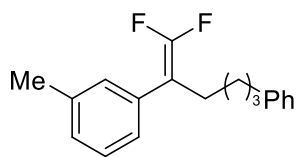
1-(1,1-difluoro-6-phenylhex-1-en-2-yl)-4-methylbenzene

(3al) : According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL, 0.9 mmol), and

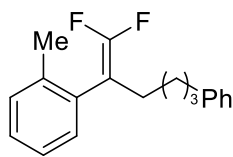
then trifluoromethyl alkene **2l** (67.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL), **[Ir(dtbbPy)(PPy)₂](PF₆)** (4.1 mg, 0.0045 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 65 mg (76% yield) of **3al** as a colorless oil: **¹H NMR (400 MHz, CDCl₃)** δ: 7.29 (t, *J* = 7.7 Hz, 2H), 7.24-7.15 (m, 7H), 2.60 (t, *J* = 7.8 Hz, 2H), 2.46-2.41 (m, 2H), 2.38 (s, 3H), 1.69-1.61 (m, 2H), 1.48-1.41 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ: 153.5 (dd, *J* = 287.5 Hz, 285.1 Hz), 142.4, 136.9, 130.6, 129.1, 128.3, 128.2, 128.1 (t, *J* = 3.4 Hz), 125.7, 92.0 (dd, *J* = 20.1 Hz, 14.4 Hz), 35.5, 30.7, 27.4, 27.2, 21.1; **¹⁹F NMR (376 MHz, CDCl₃)** δ: -92.07, -92.10; **HRMS m/z (ESI)** calcd for C₁₉H₂₀F₂ (M+H)⁺ 287.1611, found 287.1613.



1-(1,1-difluoro-6-phenylhex-1-en-2-yl)-4-methoxybenzene (3am) : According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μ L, 0.9 mmol), and then trifluoromethyl alkene **2m** (72.7 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL), **[Ir(dtbbPy)(PPy)₂](PF₆)** (4.1 mg, 0.0045 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 60 mg (66% yield) of **3am** as a colorless oil: **¹H NMR (400 MHz, CDCl₃)** δ : 7.31-7.16 (m, 7H), 6.94-6.90 (m, 2H), 3.84(s, 3H), 2.60 (t, $J = 7.6$ Hz, 2H), 2.44-2.40 (m, 2H), 1.69-1.61 (m, 2H), 1.48-1.40 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ : 158.6, 153.4 (t, $J = 285.6$ Hz), 142.4, 129.3 (t, $J = 3.4$ Hz), 128.3, 128.2, 125.8, 125.7, 113.8, 91.6 (dd, $J = 18.7$ Hz, 15.8 Hz), 55.2, 35.5, 30.7, 27.4. 27.2; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -92.59; **HRMS m/z (ESI)** calcd for C₁₉H₂₀F₂O (M+H)⁺ 303.1560, found 303.1562.

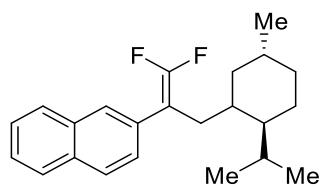


1-(1,1-difluoro-6-phenylhex-1-en-2-yl)-3-methylbenzene (3an): According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μ L, 0.9 mmol), and then trifluoromethyl alkene **2** (67.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL), **[Ir(dtbbPy)(PPy)₂](PF₆)** (4.1 mg, 0.0045 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 49 mg (57% yield) of **3an** as a colorless oil: **¹H NMR (400 MHz, CDCl₃)** δ : 7.30-7.23 (m, 3H), 7.23-7.18 (m, 1H), 7.15-7.08 (m, 5H), 2.58 (t, $J = 7.9$ Hz, 2H), 2.44-2.36 (m, 5H), 1.67-1.60 (m, 2H), 1.46-1.38 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ : 153.5 (dd, $J = 288.5$ Hz, 284.6 Hz), 142.4, 138.0, 133.6 (t, $J = 2.4$ Hz), 128.9 (t, $J = 2.9$ Hz), 128.3, 128.2, 128.0, 125.7, 125.3 (t, $J = 2.9$ Hz), 92.3 (dd, $J = 21.1$ Hz, 13.4 Hz), 35.5, 30.7, 27.5, 27.2, 21.4; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -91.72 (d, $J = 44.8$ Hz), -91.95 (d, $J = 46.2$ Hz); **HRMS m/z (ESI)** calcd for C₁₉H₂₀F₂Na (M+Na)⁺ 309.1431, found 309.1433.



1-(1,1-difluoro-4-phenylbut-1-en-2-yl)-2-methylbenzene (3ao):

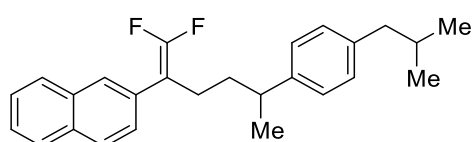
According to Method C, the reaction was carried out with alcohol **1a** (40.9 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), **dry THF** (3.0 mL), **CS₂** (54.1 μL, 0.9 mmol), and then trifluoromethyl alkene **2** (67.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), **Acetone/MeCN** (5.4 mL/0.6 mL), **[Ir(dtbbPy)(PPy)₂](PF₆)** (4.1 mg, 0.0045 mmol). The crude product was purified by flash column chromatography on silica gel (PE) to afford 31 mg (36% yield) of **3ao** as a colorless oil: ¹H NMR (400 MHz, CDCl₃) δ: 7.30-7.14 (m, 8H), 7.11-7.09 (d, *J* = 7.8 Hz, 1H), 7.15-7.08 (m, 5H), 2.59 (t, *J* = 7.9 Hz, 2H), 2.36-2.28 (m, 5H), 1.68-1.61 (m, 2H), 1.44-1.36 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 152.5 (dd, *J* = 286.5 Hz, 285.1 Hz), 142.4, 136.8, 133.2, 130.2, 129.6, 128.3, 128.2, 125.7, 90.9 (dd, *J* = 21.1 Hz, 17.3 Hz), 35.6, 31.7, 28.7, 27.0, 19.4; ¹⁹F NMR (376 MHz, CDCl₃) δ: -89.83 (d, *J* = 47.7 Hz), -94.69 (d, *J* = 46.2 Hz); HRMS *m/z* (ESI) calcd for C₁₉H₂₀F₂Na (M+Na)⁺ 309.1431, found 309.1430.



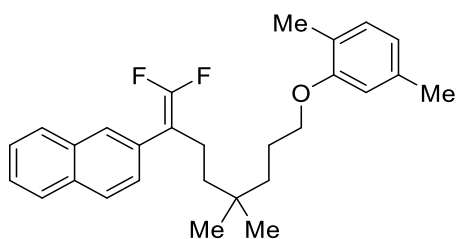
2-(1,1-difluoro-3-((2S,5S)-2-isopropyl-5-methylcyclohexyl)prop-1-en-2-yl)naphthalene (3aq) :

According to Method C, the reaction was carried out with alcohol **1ab** (31.3 mg, 0.2 mmol), **KO^tBu** (24.7 mg, 0.22 mmol), **dry THF** (2.0 mL), **CS₂** (36.0 μL, 0.6 mmol), and then trifluoromethyl alkene **2a** (53.3 mg, 0.24 mmol), **PhPCy₂** (65.9 mg, 0.24 mmol), **Acetone/MeCN** (3.6 mL/0.4 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 52 mg (76% yield, dr = 3:1) of **3aq** as a colorless oil. Data of one isomer: ¹H NMR (400 MHz, CDCl₃) δ: 7.87-7.79 (m, 4H), 7.5-7.44 (m, 3H), 2.93-2.87 (m, 1H), 2.16-2.05 (m, 1H), 1.83-1.58 (m, 3H), 1.31-1.08 (m, 2H), 1.02-0.94 (m, 1H), 0.92-0.89 (m, 3H), 0.86 (d, *J* = 6.5 Hz, 3H), 0.82-0.72 (m, 3H), 0.67 (d, *J* = 6.8 Hz, 2H), 0.64-0.56 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 154.1 (dd, *J* = 288.5 Hz, 284.6 Hz), 133.3, 132.4, 131.3 (t, *J* = 3.8 Hz), 127.9, 127.6, 127.3 (t, *J* = 3.4 Hz), 126.2, 126.0, 91.4 (dd, *J* = 21.1 Hz, 13.4 Hz), 47.6, 40.5, 36.8, 35.2, 32.4, 31.2, 26.6, 24.1, 22.8, 21.5, 15.2; ¹⁹F NMR (376 MHz, CDCl₃) δ: -91.01 (d, *J* = 43.3 Hz), -91.25 (d, *J* = 43.3 Hz). Data of the other isomer: ¹H NMR (400 MHz, CDCl₃) δ: 7.87-7.79 (m, 4H), 7.5-7.44 (m, 3H), 2.93-2.87 (m, 1H), 2.16-2.05 (m, 1H), 1.83-1.58 (m, 3H), 1.31-1.08 (m, 2H), 1.02-0.94 (m, 1H), 0.92-0.89 (m, 3H), 0.86 (d, *J* = 6.5 Hz, 3H),

0.82-0.72 (m, 3H), 0.67 (d, $J = 6.8$ Hz, 2H), 0.64-0.56 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 154.0 (dd, $J = 288.5$ Hz, 284.6 Hz), 133.2, 132.5, 127.8, 127.6, 126.3, 91.8 (dd, $J = 22.0$ Hz, 13.6 Hz), 45.0, 37.8, 35.8, 32.8, 29.3, 26.0, 25.1, 23.7, 21.7, 20.6; ^{19}F NMR (376 MHz, CDCl_3) δ : -91.88 (d, $J = 44.8$ Hz), -92.24 (d, $J = 44.8$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{23}\text{H}_{28}\text{F}_2$ ($\text{M}+\text{H}$) $^+$ 343.2237, found 343.2239.

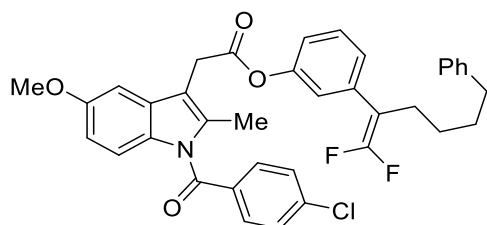


(S)-2-(1,1-difluoro-5-(4-isobutylphenyl)hex-1-en-2-yl)naphthalene (3ar) : According to Method C, the reaction was carried out with alcohol **1ac** (38.4 mg, 0.2 mmol), **KO^tBu** (24.7 mg, 0.22 mmol), **dry THF** (2.0 mL), **CS₂** (36.0 μL , 0.6 mmol), and then trifluoromethyl alkene **2a** (53.3 mg, 0.24 mmol), **PhPCy₂** (65.9 mg, 0.24 mmol), **Acetone/MeCN** (3.6 mL/0.4 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 56 mg (74% yield) of **3ar** as a colorless oil: ^1H NMR (400 MHz, CDCl_3) δ : 7.86-7.79 (m, 3H), 7.68 (s, 1H), 7.53-7.48 (m, 2H), 7.40 (tt, $J = 1.7$ Hz, 1.8 Hz, 1H), 7.08 (d, $J = 4.7$ Hz, 4H), 2.74-2.69 (m, 1H), 2.49 (d, $J = 7.2$ Hz, 3H), 2.45-2.35 (m, 2H), 1.92-1.86 (m, 1H), 1.73-1.66 (m, 2H), 1.24 (d, $J = 7.0$ Hz, 3H), 0.95 (d, $J = 6.5$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ : 153.6 (dd, $J = 289.4$ Hz, 285.6 Hz), 144.0, 139.3, 133.2, 132.4, 131.0 (t, $J = 3.8$ Hz), 129.1, 127.9, 127.5, 127.1 (t, $J = 3.4$ Hz), 126.7, 126.2, 126.1 (t, $J = 3.4$ Hz), 126.0, 92.5 (dd, $J = 21.6$ Hz, 12.5 Hz), 45.0, 39.2, 36.3, 30.2, 25.8, 22.4, 22.3; ^{19}F NMR (376 MHz, CDCl_3) δ : -90.78 (d, $J = 41.9$ Hz), -91.29 (d, $J = 43.3$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{26}\text{H}_{28}\text{F}_2$ ($\text{M}+\text{H}$) $^+$ 379.2237, found 379.2240.



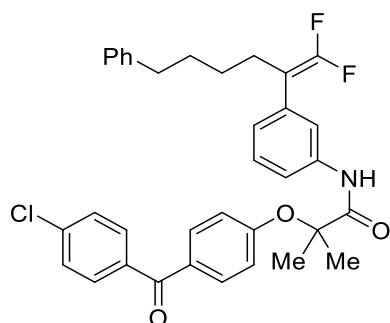
2-(8-(2,5-dimethylphenoxy)-1,1-difluoro-5,5-dimethyloct-1-en-2-yl)naphthalene (3as) : According to Method C, the reaction was carried out with alcohol **1ad** (47.2 mg, 0.2 mmol), **KO^tBu** (24.7 mg, 0.22 mmol), **dry THF** (2.0 mL), **CS₂** (36.0 μL , 0.6 mmol), and then trifluoromethyl alkene **2a** (53.3 mg, 0.24 mmol), **PhPCy₂** (65.9 mg, 0.24 mmol), **Acetone/MeCN** (3.6 mL/0.4 mL). The crude product was purified by flash column chromatography on silica gel (PE) to afford 40

mg (47% yield) of **3as** as a colorless oil: $^1\text{H NMR}$ (400 MHz, CDCl_3) δ : 7.87-7.80 (m, 4H), 7.52-7.46 (m, 3H), 7.05 (d, $J = 7.4$ Hz, 1H), 6.70-6.65 (m, 2H), 3.93 (t, $J = 6.3$ Hz, 2H), 2.53-2.47 (m, 2H), 2.34 (s, 3H), 2.21 (s, 3H), 1.76-1.68 (m, 2H), 1.44-1.35 (m, 4H), 0.94 (s, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ : 157.1, 153.5 (dd, $J = 288.9$ Hz, 285.6 Hz), 136.4, 133.3, 132.4, 131.2 (t, $J = 3.4$ Hz), 130.3, 128.0, 127.9, 127.6, 127.1 (t, $J = 3.4$ Hz), 126.2, 126.0, 126.0 (t, $J = 3.4$ Hz), 123.5, 120.6, 111.9, 93.1 (dd, $J = 21.1$ Hz, 12.9 Hz), 68.4, 39.8, 37.6, 32.5, 27.0, 24.2, 22.6, 21.4, 15.8; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ : -91.32 (d, $J = 43.3$ Hz), -91.54 (d, $J = 43.4$ Hz); **HRMS** m/z (ESI) calcd for $\text{C}_{28}\text{H}_{32}\text{F}_2\text{O}$ ($\text{M}+\text{H}$) $^+$ 423.2499, found 423.2498.

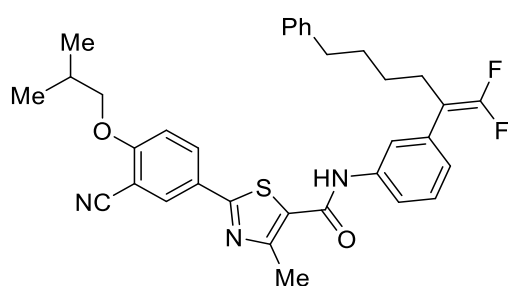


3-(1,1-difluoro-6-phenylhex-1-en-2-yl)phenyl-2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate (3at) : According to Method C, the reaction was carried out with alcohol **1a** (27.2 mg, 0.2 mmol), **KO^tBu** (24.7

mg, 0.22 mmol), **dry THF** (2.0 mL), **CS₂** (36.0 μL , 0.6 mmol), and then trifluoromethyl alkene **2q** (126.5 mg, 0.24 mmol), **PhPCy₂** (65.9 mg, 0.24 mmol), **Acetone/MeCN** (3.6 mL/0.4 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 5:1) to afford 83 mg (66% yield) of **3at** as a green oil: $^1\text{H NMR}$ (400 MHz, CDCl_3) δ : 7.70 (d, $J = 8.6$ Hz, 2H), 7.49 (d, $J = 8.6$ Hz, 2H), 7.36-7.30 (m, 1H), 7.28-7.24 (m, 2H), 7.19-7.12 (m, 4H), 7.08-6.99 (m, 3H), 6.92 (d, $J = 9.0$ Hz, 1H), 6.72 (d, $J = 2.6$ Hz, 1H), 3.93 (s, 2H), 3.84 (s, 3H), 2.57 (t, $J = 7.8$ Hz, 2H), 2.47 (s, 3H), 2.43-2.38 (m, 2H), 1.66-1.58 (m, 2H), 1.45-1.38 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ : 169.2, 168.3, 156.1, 153.7 (dd, $J = 291.2$ Hz, 286.5 Hz), 150.7, 142.3, 139.3, 136.2, 135.2, 133.8, 131.2, 130.8, 130.5, 129.3, 129.1, 128.3, 128.2, 125.8, 125.7, 121.2 (t, $J = 3.8$ Hz), 120.2, 115.0, 111.9, 111.8, 101.2, 91.7 (dd, $J = 20.6$ Hz, 13.9 Hz), 55.1, 35.4, 30.6, 30.5, 27.2, 13.4; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ : -90.33, -90.36; **HRMS** m/z (ESI) calcd for $\text{C}_{37}\text{H}_{32}\text{ClF}_2\text{NO}_4$ ($\text{M}+\text{H}$) $^+$ 628.2066, found 628.2062.

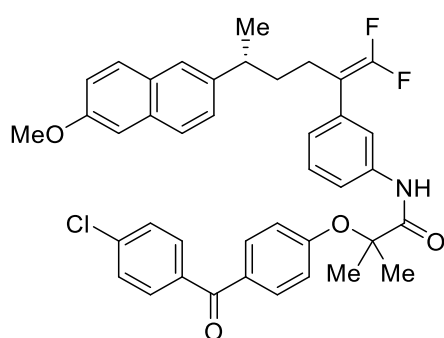


2-(4-(4-chlorobenzoyl)phenoxy)-N-(3-(1,1-difluoro-6-phenylhex-1-en-2-yl)phenyl)-2-methylpropanamide (3au) : According to Method C, the reaction was carried out with alcohol **1a** (27.2 mg, 0.2 mmol), **KO^tBu** (24.7 mg, 0.22 mmol), **dry THF** (2.0 mL), **CS₂** (36.0 μ L, 0.6 mmol), and then trifluoromethyl alkene **2r** (120.0 mg, 0.24 mmol), **PhPCy₂** (65.9 mg, 0.24 mmol), **Acetone/MeCN** (3.6 mL/0.4 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 5:1) to afford 90 mg (79% yield) of **3au** as a green solid: **¹H NMR (400 MHz, CDCl₃)** δ : 8.32 (s, 1H), 7.77 (dd, J = 8.8 Hz, 8.7 Hz, 4H), 7.51-7.44 (m, 4H), 7.32 (t, J = 8.0 Hz, 1H), 7.27-7.23 (m, 2H), 7.17-7.11 (m, 3H), 7.08 (d, J = 8.8 Hz, 3H), 2.57 (t, J = 7.8 Hz, 2H), 2.44-2.39 (m, 2H), 1.69 (s, 6H), 1.66-1.58 (m, 2H), 1.45-1.38 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ : 194.1, 172.2, 158.1, 153.6 (dd, J = 289.0 Hz, 285.6 Hz), 142.3, 138.7, 137.4, 135.9, 134.7 (t, J = 2.9 Hz), 132.1, 131.9, 131.2, 129.1, 128.6, 128.3, 128.2, 125.6, 124.6 (t, J = 3.4 Hz), 120.2, 119.7 (t, J = 3.4 Hz), 118.8, 92.0 (dd, J = 22.0 Hz, 12.9 Hz), 82.4, 35.4, 3.6, 27.3, 27.1, 25.0; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -90.94 (d, J = 43.4 Hz), -91.19 (d, J = 43.3 Hz); **HRMS m/z (ESI)** calcd for C₃₅H₃₂ClF₂NO₃ (M+H)⁺ 588.2117, found 588.2114.



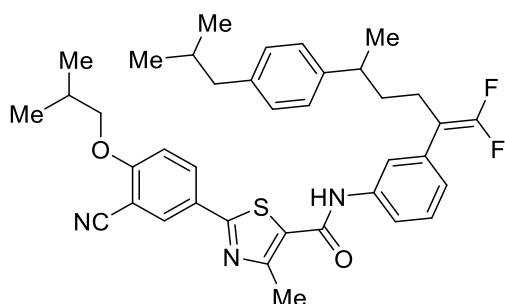
2-(3-cyano-4-isobutoxyphenyl)-N-(3-(1,1-difluoro-6-phenylhex-1-en-2-yl)phenyl)-4-methylthiazole-5-carboxamide (3av) : According to Method C, the reaction was carried out with alcohol **1a** (27.2 mg, 0.2 mmol), **KO^tBu** (24.7 mg, 0.22 mmol), **dry THF** (2.0 mL), **CS₂** (36.0 μ L, 0.6 mmol), and then trifluoromethyl alkene **2s** (116.4 mg, 0.24 mmol), **PhPCy₂** (65.9 mg, 0.24 mmol), **Acetone/MeCN** (3.6 mL/0.4 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 3:1) to afford 70 mg (60% yield) of **3av** as a yellow solid: **¹H NMR (400 MHz, CDCl₃)** δ : 8.12 (d, J = 2.3 Hz, 1H), 8.06 (dd, J = 2.4 Hz, 2.5 Hz, 1H), 7.64 (s, 1H), 7.54 (d, J = 6.6 Hz, 2H), 7.35 (t, J = 8.8 Hz, 1H), 7.26 (t, J = 7.6 Hz, 2H), 7.18-7.09 (m, 4H), 7.02 (d, J = 8.9 Hz, 1H), 3.90 (d, J = 6.5 Hz, 2H), 2.78 (s, 3H), 2.58 (t, J =

7.8 Hz, 2H), 2.46-2.41 (m, 2H), 2.23-2.16 (m, 1H), 1.67-1.59 (m, 3H), 1.47-1.39 (m, 2H), 1.10 (d, $J = 6.8$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ : 164.9, 162.4, 159.8, 157.1, 153.6 (dd, $J = 288.9$ Hz, 285.6 Hz), 142.3, 137.5, 134.8, 132.5, 131.9, 129.2, 128.3, 128.2, 125.7, 125.6, 124.9, 120.1, 119.3, 115.4, 112.6, 102.8, 92.0 (dd, $J = 22.1$ Hz, 12.9 Hz), 75.7, 35.4, 30.6, 28.1, 27.3, 27.2, 19.0, 17.5; ^{19}F NMR (376 MHz, CDCl_3) δ : -90.78 (d, $J = 43.3$ Hz), -91.03 (d, $J = 41.9$ Hz); HRMS m/z (ESI) calcd for $\text{C}_{34}\text{H}_{33}\text{F}_2\text{N}_3\text{O}_2\text{S}$ ($\text{M}+\text{H}$) $^+$ 586.2340, found 586.2343.



(R)-2-(4-(4-chlorobenzoyl)phenoxy)-N-(3-(1,1-difluoro-5-(6-methoxynaphthalen-2-yl)hex-1-en-2-yl)phenyl)-2-methylpropanamide (3aw) :

According to Method C, the reaction was carried out with alcohol **1ae** (43.2 mg, 0.2 mmol), **KO^tBu** (24.7 mg, 0.22 mmol), **dry THF** (2.0 mL), **CS₂** (36.0 μL , 0.6 mmol), and then trifluoromethyl alkene **2r** (120.0 mg, 0.24 mmol), **PhPCy₂** (65.9 mg, 0.24 mmol), **Acetone/MeCN** (3.6 mL/0.4 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 10:1) to afford 47 mg (35% yield) of **3aw** as a colorless oil: ^1H NMR (400 MHz, CDCl_3) δ : 8.30 (s, 1H), 7.78 (d, $J = 8.8$ Hz, 2H), 7.72 (d, $J = 8.5$ Hz, 2H), 7.66 (dd, $J = 2.5$ Hz, 2.6 Hz, 2H), 7.50-7.43 (m, 5H), 7.29 (t, $J = 7.9$ Hz, 1H), 7.25 (dd, $J = 1.8$ Hz, 1.8 Hz, 1H), 7.13-7.05 (m, 4H), 7.00 (d, $J = 8.1$ Hz, 1H), 3.90 (s, 3H), 2.84-2.79 (m, 1H), 2.38-2.23 (m, 2H), 1.77-1.70 (m, 2H), 1.68 (s, 6H), 1.28 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 194.2, 172.2, 158.1, 157.2, 156.3, 153.4 (t, $J = 287.0$ Hz), 141.8, 138.7, 137.4, 135.9, 134.7, 133.2, 132.2, 132.0, 131.2, 129.1, 129.0, 128.6, 126.9, 126.0, 125.1, 124.5, 120.2, 119.7 (t, $J = 3.4$ Hz), 118.8, 118.6, 105.6, 92.1 (t, $J = 17.3$ Hz), 82.4, 55.3, 39.4, 36.0, 25.9, 25.0, 22.0; ^{19}F NMR (376 MHz, CDCl_3) δ : -90.77; HRMS m/z (ESI) calcd for $\text{C}_{40}\text{H}_{36}\text{ClF}_2\text{NO}_4$ ($\text{M}+\text{H}$) $^+$ 668.2379, found 668.2377.



(R)-2-(3-cyano-4-isobutoxyphenyl)-N-(3-(1,1-difluoro-5-(4-isobutylphenyl)hex-1-en-2-yl)phenyl)-4-methylthiazole-5-carboxamide (3ax) : According to Method C, the reaction was carried out with alcohol **1ac** (38.4 mg, 0.2 mmol), **KO^tBu** (24.7 mg, 0.22 mmol), **dry THF** (2.0 mL), **CS₂** (36.0

μL , 0.6 mmol), and then trifluoromethyl alkene **2s** (116.4 mg, 0.24 mmol), **PhPCy₂** (65.9 mg, 0.24 mmol), **Acetone/MeCN** (3.6 mL/0.4 mL). The crude product was purified by flash column chromatography on silica gel (PE:EA = 5:1) to afford 58 mg (45% yield) of **3ax** as a white solid: **¹H NMR (400 MHz, CDCl₃)** δ : 8.12 (m, 2H), 7.64-7.53 (m, 2H), 7.44 (s, 1H), 7.32 (t, $J = 8.0$ Hz, 1H), 7.04-7.00 (m, 6H), 3.91 (d, $J = 6.5$ Hz, 2H), 2.78 (s, 3H), 2.68-2.63 (m, 1H), 2.42 (d, $J = 7.2$ Hz, 2H), 2.36-2.17 (m, 3H), 1.86-1.79 (m, 1H), 1.67-1.60 (m, 2H), 1.22 (d, $J = 6.9$ Hz, 3H), 1.10 (d, $J = 6.8$ Hz, 6H), 0.89 (d, $J = 6.6$ Hz, 6H); **¹³C NMR (100 MHz, CDCl₃)** δ : 164.9, 162.5, 159.7, 156.9, 153.4 (t, $J = 287.0$ Hz), 143.9, 139.3, 137.5, 134.7, 132.5, 131.9, 129.1, 129.0, 126.6, 125.7, 125.6, 124.8, 120.1, 119.2, 115.4, 112.6, 102.9, 92.1 (dd, $J = 18.7$ Hz, 15.8 Hz), 45.0, 39.1, 36.2, 30.2, 28.1, 25.7, 22.3, 22.2, 19.0, 17.5; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -90.66; **HRMS m/z (ESI)** calcd for C₃₈H₄₁F₂N₃O₂S (M+H)⁺ 642.2966, found 642.2964.

6. Gram Scale Reaction

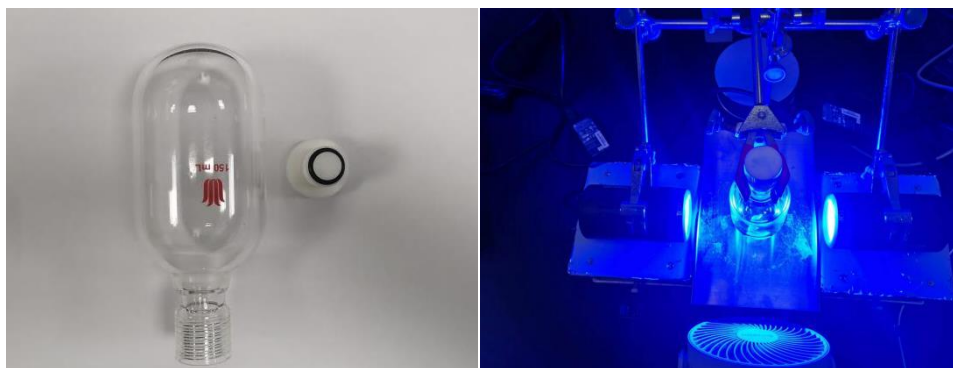
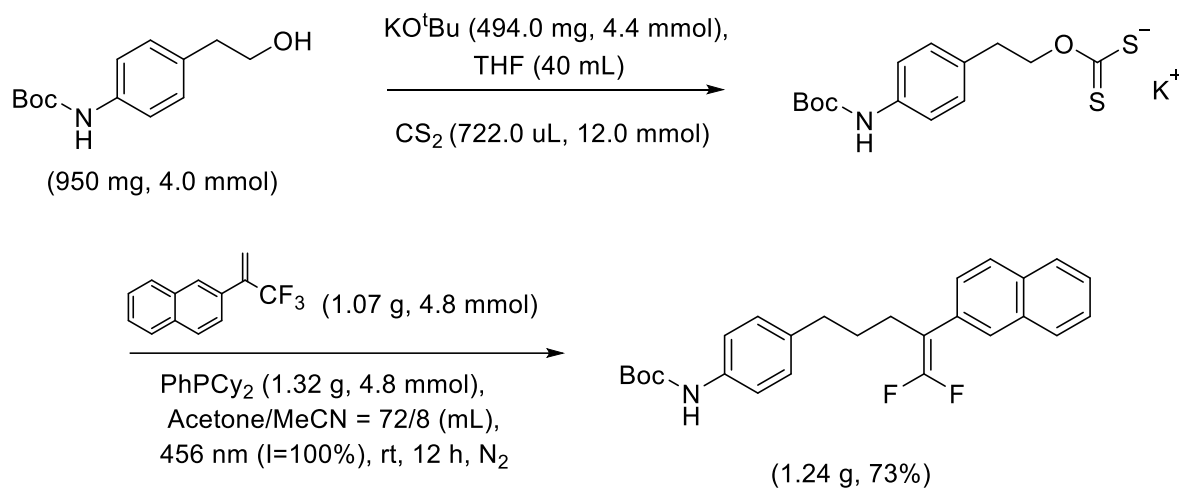


Figure S2. An over-dried 150 mL pressure tubing (left). Photoreaction setup (right).



In a N₂-filled glovebox, an oven-dried 150.0 mL pressure tubing equipped with a magnetic stir bar was charged sequentially with alcohol **1c** (950 mg, 4.0 mmol), **KO^tBu** (494.0 mg, 4.4 mmol), and **dry THF** (40.0 mL). After being sealed with a septum cap and transferred out of the glovebox, the reaction mixture was stirred at room temperature for 30 minutes, followed by the addition of **CS₂** (722.0 μL, 12.0 mmol) via microsyringe at 0°C and continued to be stirred for 3 hours at 0°C before removing the solvent in vacuo. The system was transferred into the glovebox, then **2a** (1.07 g, 4.8 mmol), **PhPCy₂** (1.32 g, 4.8 mmol), and the mixed solvents of **Acetone/MeCN** (72 mL/8 mL) were added. The vial was sealed with a septum cap and transferred out of the glovebox. The reaction mixture was stirred at room temperature under blue LEDs (456 nm, the light intensity is 100%) irradiation for 12 h (the reaction tube placed approximately ~ 6 cm from the bulbs and using a fan to keep at room temperature). The solvent was removed in vacuo and the residue was

purified by flash column chromatography on silica gel (PE:EA = 20:1) to afford 1.24 g (73% yield) of **3c**.

7. Cyclic voltammetry of **2l** and **2m**

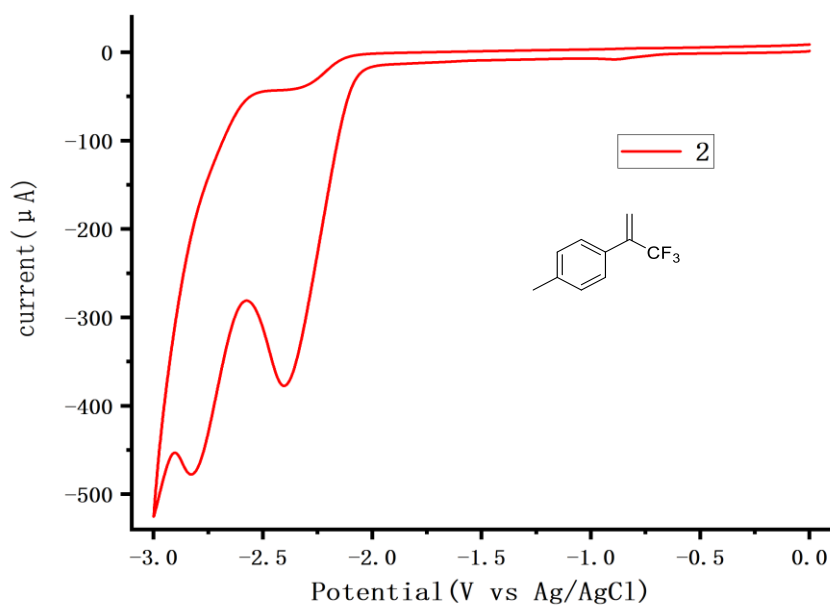


Figure S3. Cyclic voltammetry of 1-methyl-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene

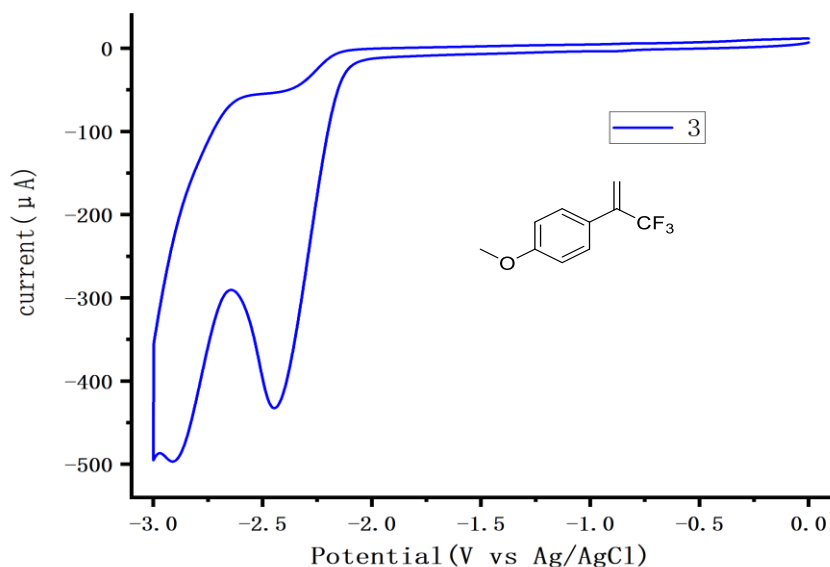
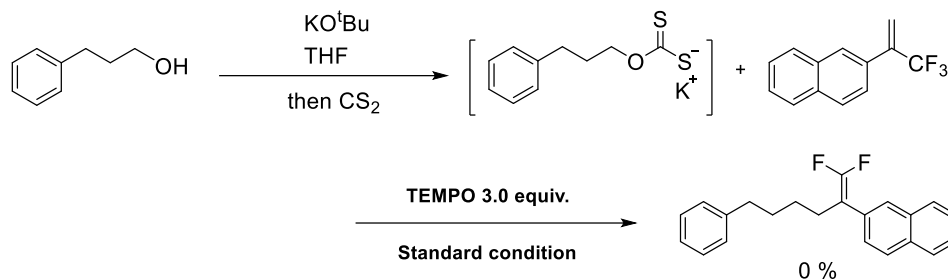


Figure S4. Cyclic voltammetry of
1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene

Cyclic voltammetry of alkenes in DMF. Test conditions: A cyclic voltammograms in solvent (10 mL) by using glassy carbon as the working electrode, Pt wire as the counter electrode and Ag/AgCl as the reference electrode under N₂ at room temperature. 10 mL of DMF containing 0.1 M TEAB was poured into the electrochemical cell in all experiments. The scan rate is 0.1 V/s. The reduction potential of 1-methyl-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene was observed at -2.41 V. The reduction potential of 1-methoxy-4-(3,3,3-trifluoroprop-1-en-2-yl)benzene was observed at -2.45 V.

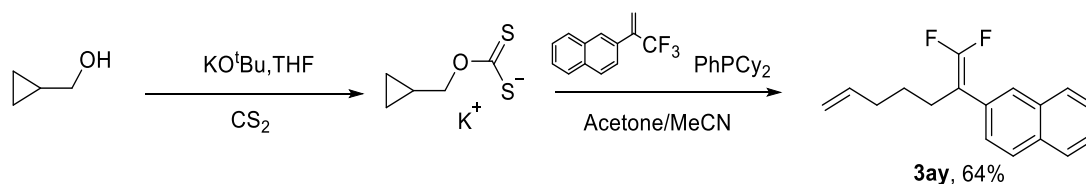
8. Mechanistic Studies

Radical trapping experiment



In a nitrogen-filled glovebox, over-dried 20.0 mL vial with a stirring bar was added, with alcohol **1a** (27.2 mg, 0.2 mmol), **KO^tBu** (24.7 mg, 0.22 mmol), and **dry THF** (2.0 mL). After being sealed with a septum cap and transferred out of the glovebox, the reaction mixture was stirred at room temperature for 30 minutes, followed by the addition of **CS₂** (36.1 μ L, 0.6 mmol) via microsyringe at 0°C and continued to be stirred for 3 hours at 0°C before removing the solvent in vacuo. The system was transferred into the glovebox, then the reaction bottle was added **olefin 2a** (53.3 mg, 0.24 mmol), **PhPCy₂** (65.9 mg, 0.24 mmol), **TEMPO** (93.8 mg, 0.6 mmol) and **Acetone/MeCN** = 3.6/0.4 (mL). The resulting mixture was stirred at room temperature under blue LEDs (λ = 456 nm, I = 25%) irradiation for 12 h. After this time, the reaction mixture was concentrated under reduced pressure. The yield of **3a** was 0% determined by ¹⁹F NMR using 1, 4-Difluorobenzene as an internal standard.

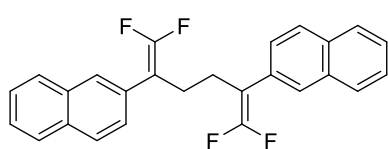
Radical clock experiment



In a N₂-filled glovebox, an oven-dried 20 mL glass vial equipped with a magnetic stir bar was charged sequentially with **cyclopropylmethanol** (21.6 mg, 0.3 mmol), **KO^tBu** (37.0 mg, 0.33 mmol), and **dry THF** (3.0 mL). After being sealed with a septum cap and transferred out of the glovebox, the reaction mixture was stirred at room temperature for 30 minutes, followed by the addition of **CS₂** (54.1 μ L, 0.9 mmol) via microsyringe at 0°C and continued to be stirred for 3 hours at 0°C before

removing the solvent in vacuo. The system was transferred into the glovebox, then **2a** (80.0 mg, 0.36 mmol), **PhPCy₂** (98.8 mg, 0.36 mmol), and the mixed solvents of **Acetone/MeCN** (5.4 mL/0.6 mL) were added. The resulting mixture was stirred at room temperature under blue LEDs ($\lambda = 456$ nm, I = 25%) irradiation for 12 h. The solvent was removed in vacuo and the residue was purified by flash column chromatography on silica gel (PE) to afford 50 mg (64% yield) of **3ay** as a colorless oil. **¹H NMR (400 MHz, CDCl₃)** δ : 7.86-7.79 (m, 4H), 7.53-7.45 (m, 3H), 5.84-5.74 (m, 1H), 5.05-4.96 (m, 2H), 2.57-2.52 (m, 2H), 2.11 (q, $J = 7.1$ Hz, 2H), 1.56-1.49 (m, 2H); **¹³C NMR (100 MHz, CDCl₃)** δ : 153.8 (dd, $J = 288.9$ Hz, 285.6 Hz), 138.1, 133.3, 132.4, 131.1 (t, $J = 3.4$ Hz), 128.0, 127.9, 127.6, 127.3 (t, $J = 3.4$ Hz), 126.2, 126.1 (t, $J = 2.9$ Hz), 126.1, 114.9, 92.4 (dd, $J = 21.6$ Hz, 12.9 Hz), 33.0, 27.1, 27.0; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -91.05 (d, $J = 43.3$ Hz), -91.33 (d, $J = 43.3$ Hz). All data are in accordance with the literature.¹⁰

Byproduct



2,2'-(1,1,6,6-tetrafluorohexa-1,5-diene-2,5-diyl)dinaphthalene (3a''): White solid. **¹H NMR (400 MHz, CDCl₃)** δ : 7.82 (dd, $J = 7.0$ Hz, 8.6 Hz, 4H), 7.67 (d, $J = 7.1$ Hz, 2H), 7.57 (s, 2H), 7.50-7.46 (m, 4H), 7.34 (d, $J = 8.6$ Hz, 2H), 2.62 (s, 4H); **¹³C NMR (100 MHz, CDCl₃)** δ : 153.9 (dd, $J = 289.9$ Hz, 286.6 Hz), 133.2, 132.4, 130.4, 128.0, 127.9, 127.6, 127.3, 126.3, 126.2, 126.0, 91.7 (dd, $J = 21.6$ Hz, 13.4 Hz), 26.3; **¹⁹F NMR (376 MHz, CDCl₃)** δ : -89.97 (d, $J = 40.5$ Hz), -90.63 (d, $J = 40.5$ Hz); **HRMS m/z (ESI)** calcd for C₂₆H₁₈F₄ (M+H)⁺ 407.1423, found 407.1421.

UV-Vis Absorption Spectra

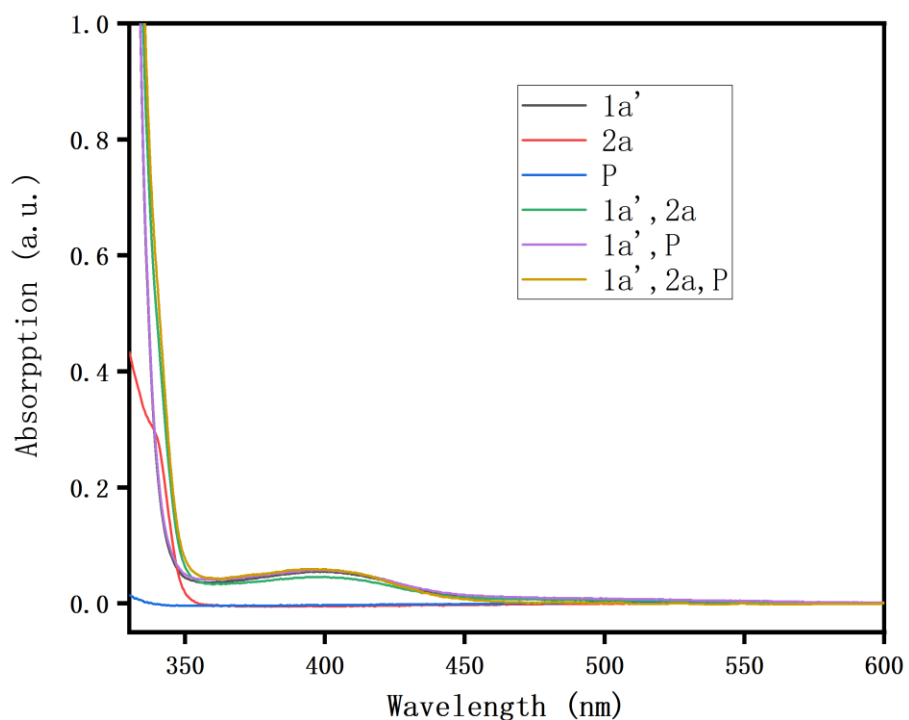


Figure S5. UV/Vis absorption spectra of single reaction component and combined ones

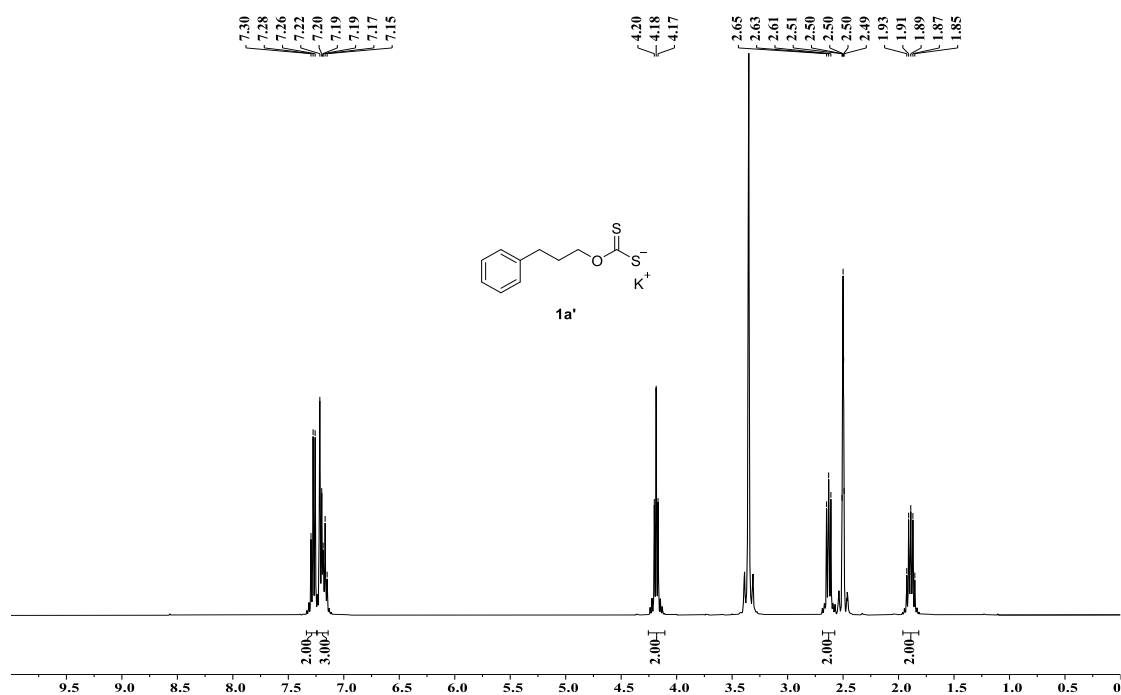
The UV-Vis absorption spectra of all solution were introduced to a 1 cm path length quartz cuvette and analyzed using a Shimadzu UV/Vis spectrophotometer UV-3600plus. **1a'**: 10^{-3} M in **Acetone/MeCN**. Olefin (**2a**): 1.2×10^{-3} M in **Acetone/MeCN**. PhPCy₂ (**P**): 1.5×10^{-3} M in **Acetone/MeCN**. These results indicated that an EDA complex is not formed.

9. References

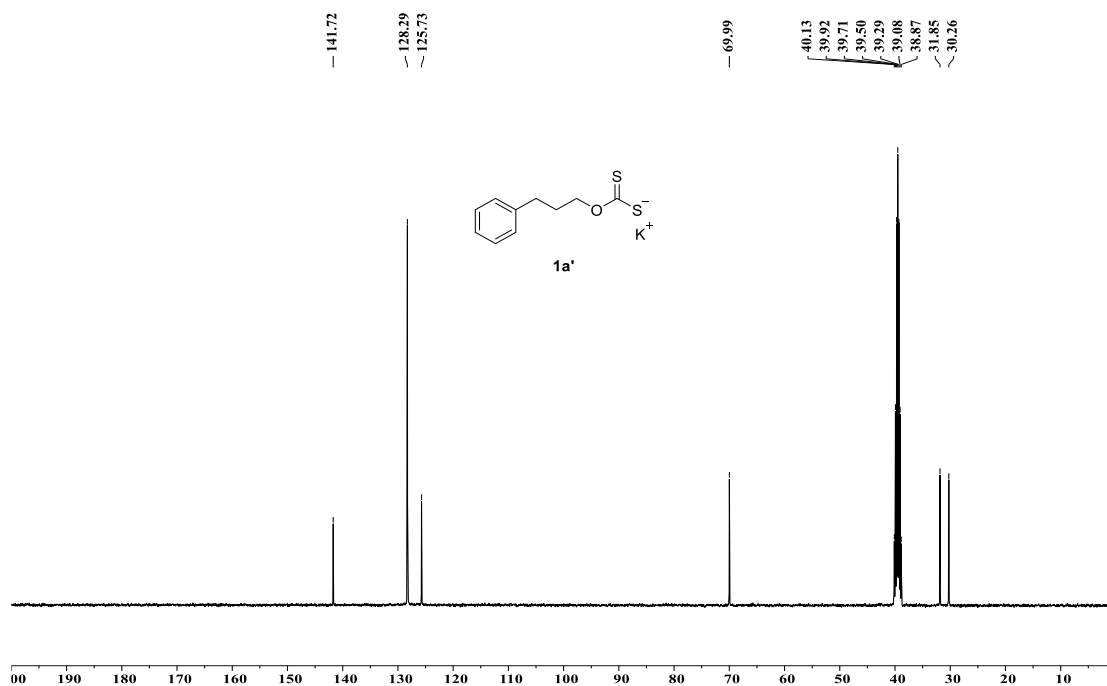
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10. Copies of NMR Spectra

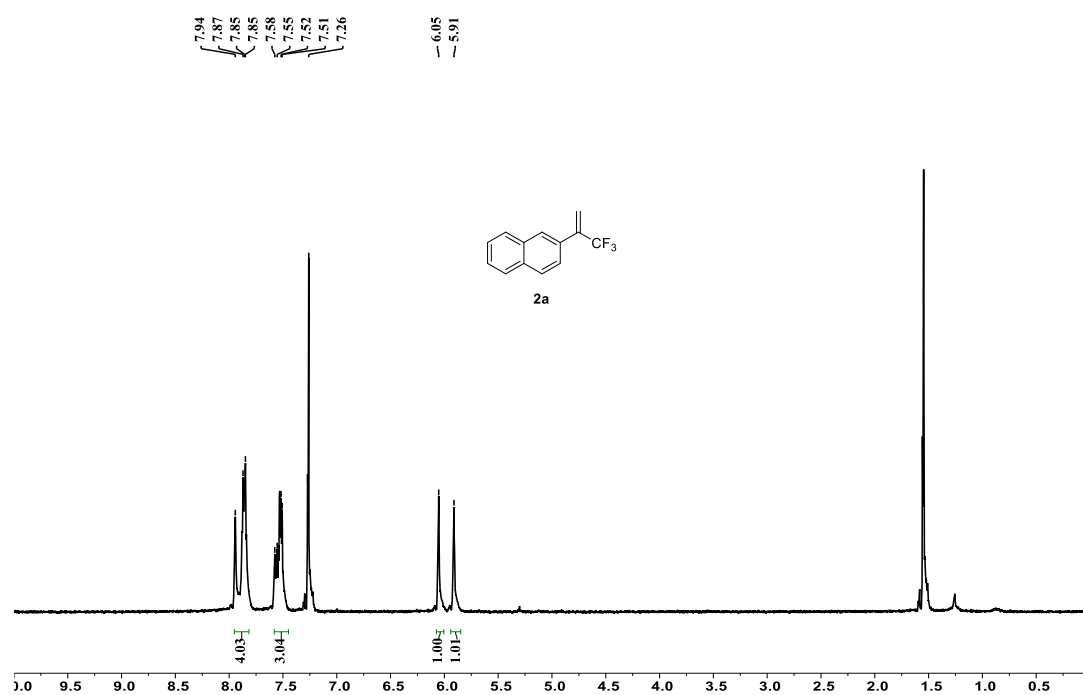
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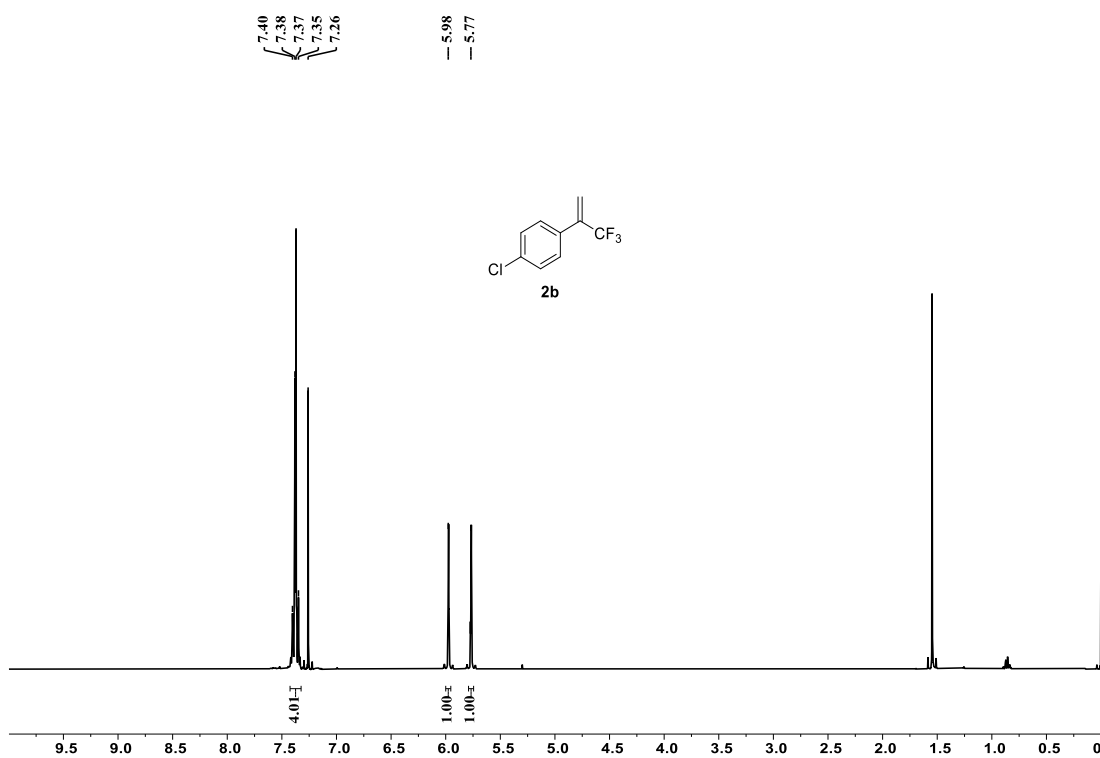
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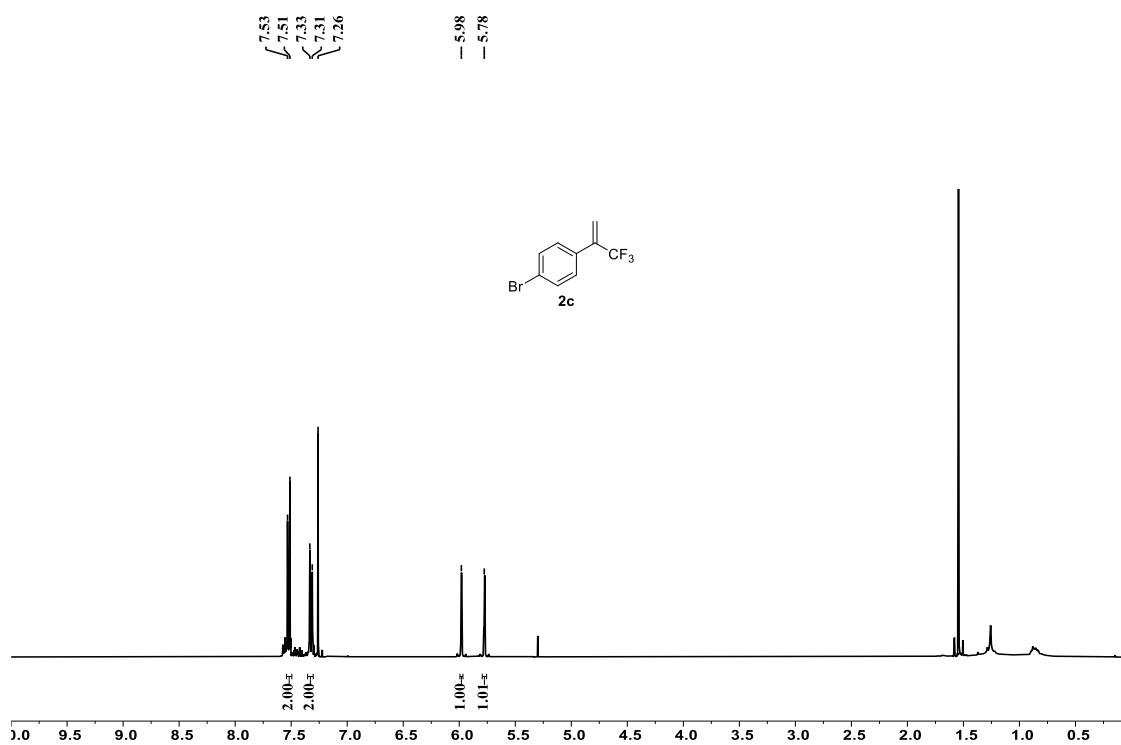
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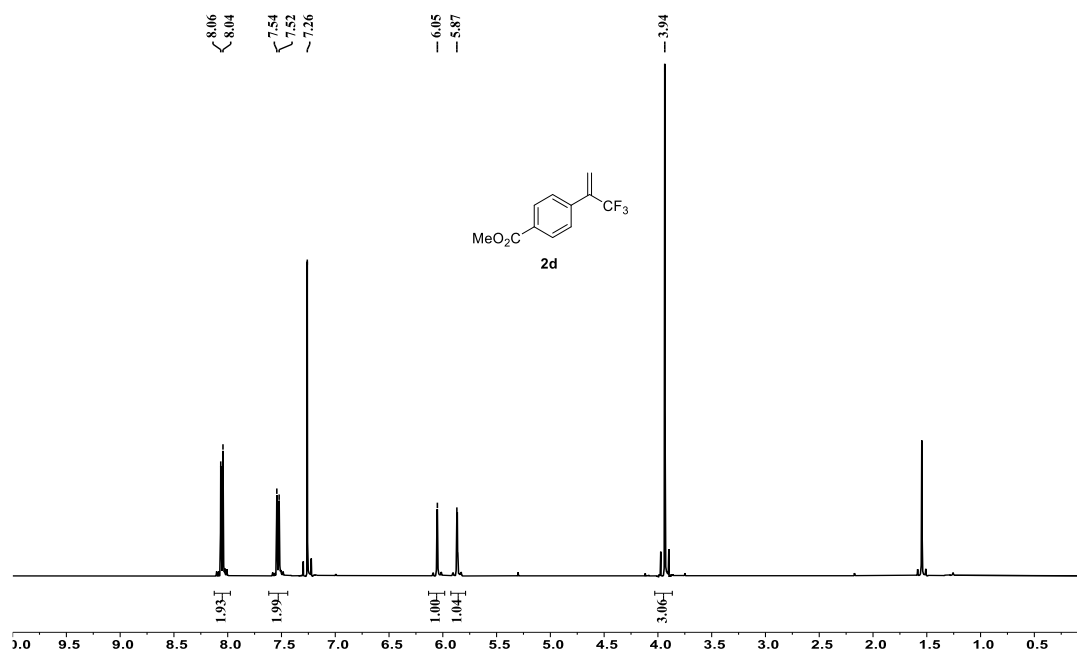
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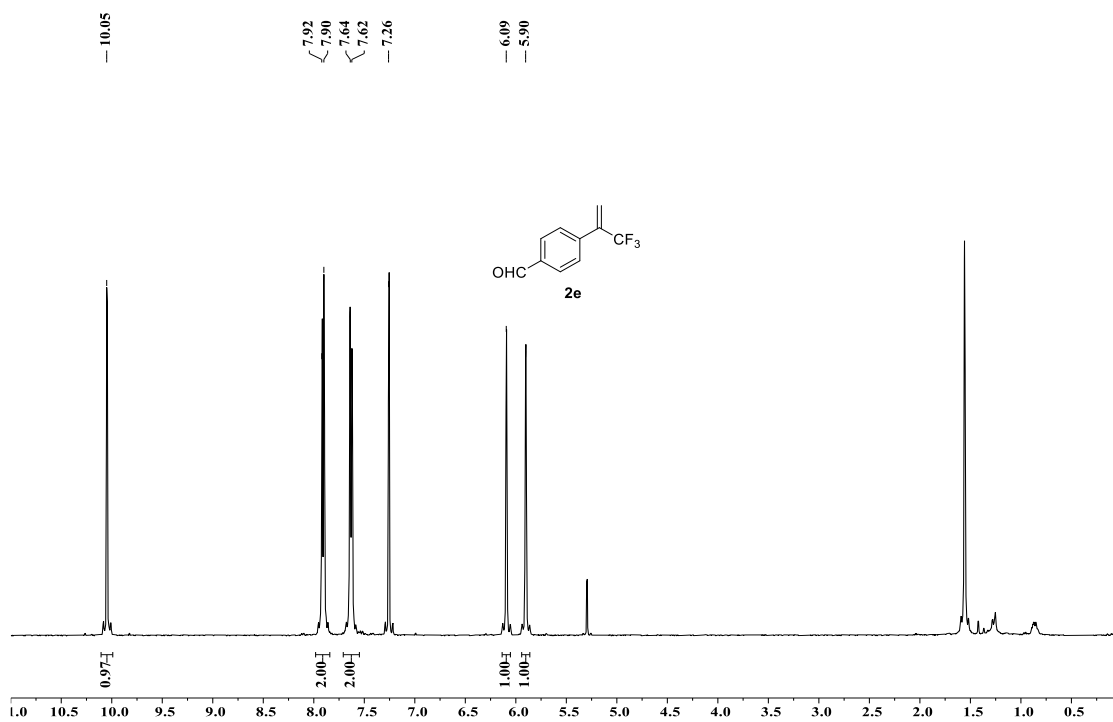
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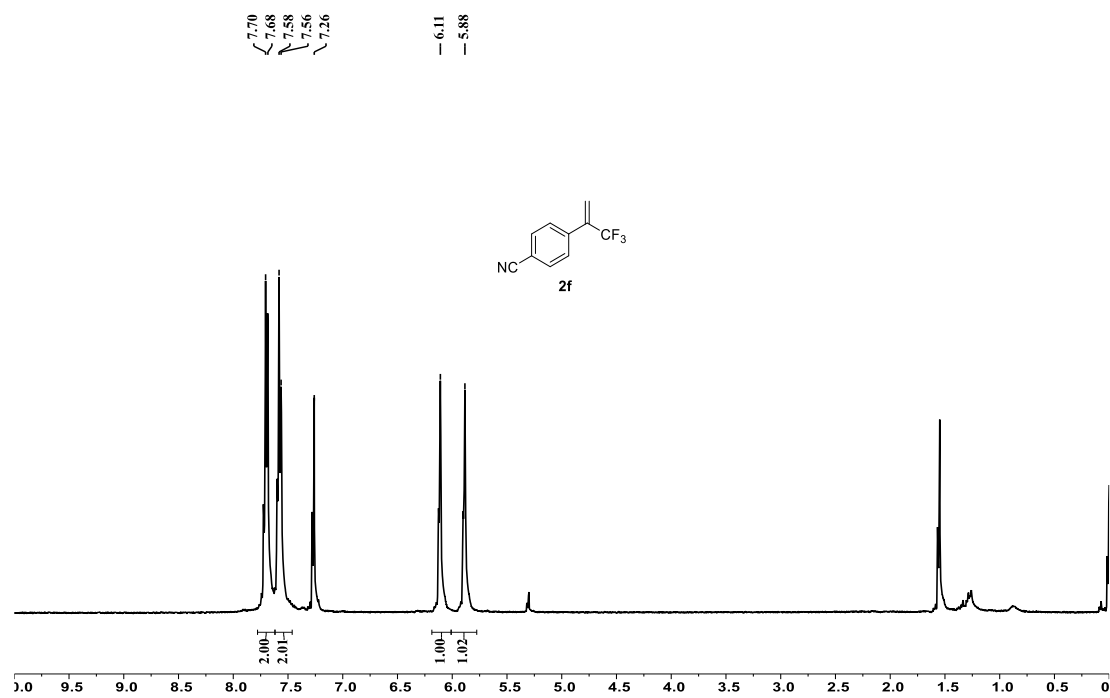
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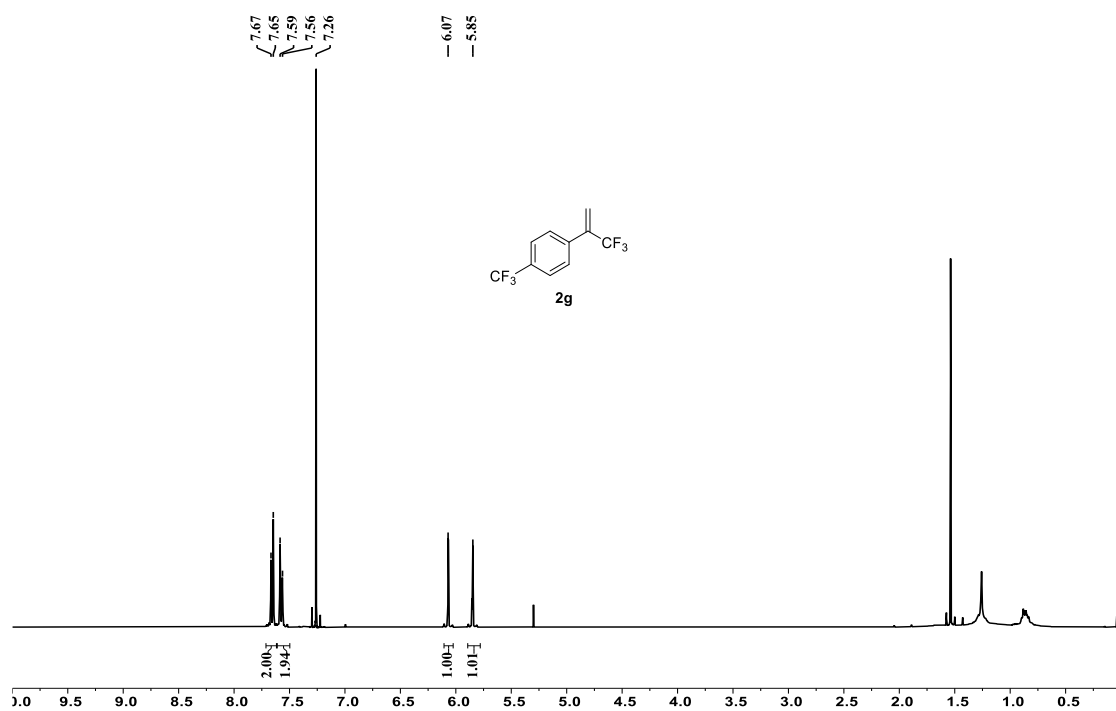
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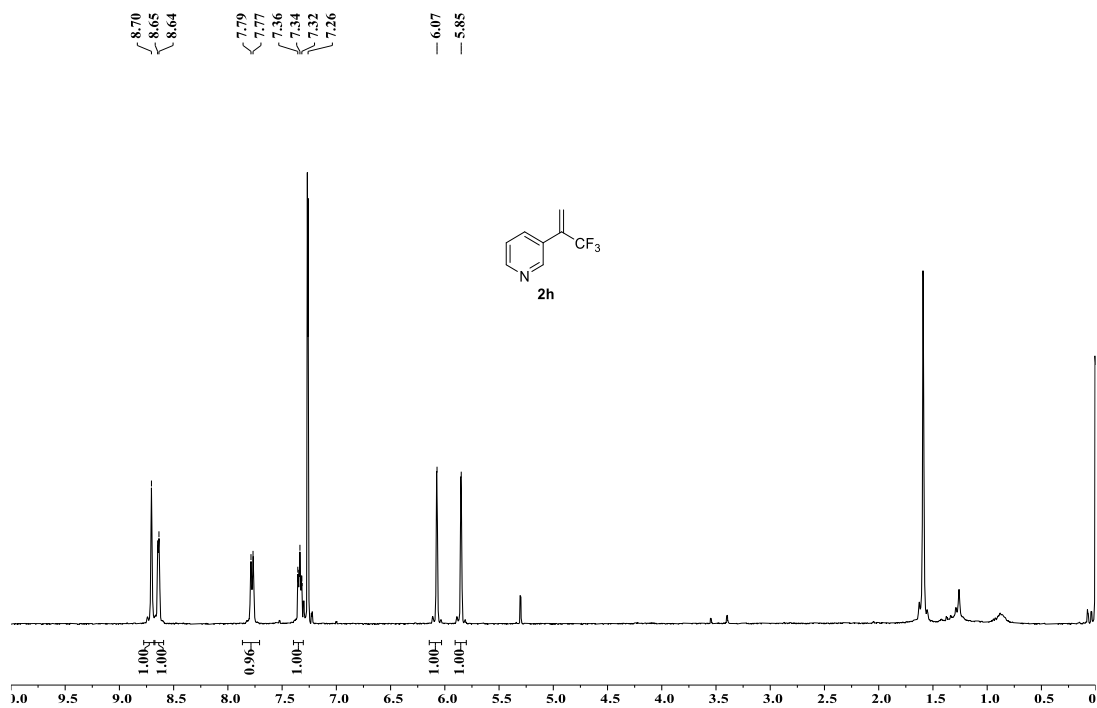
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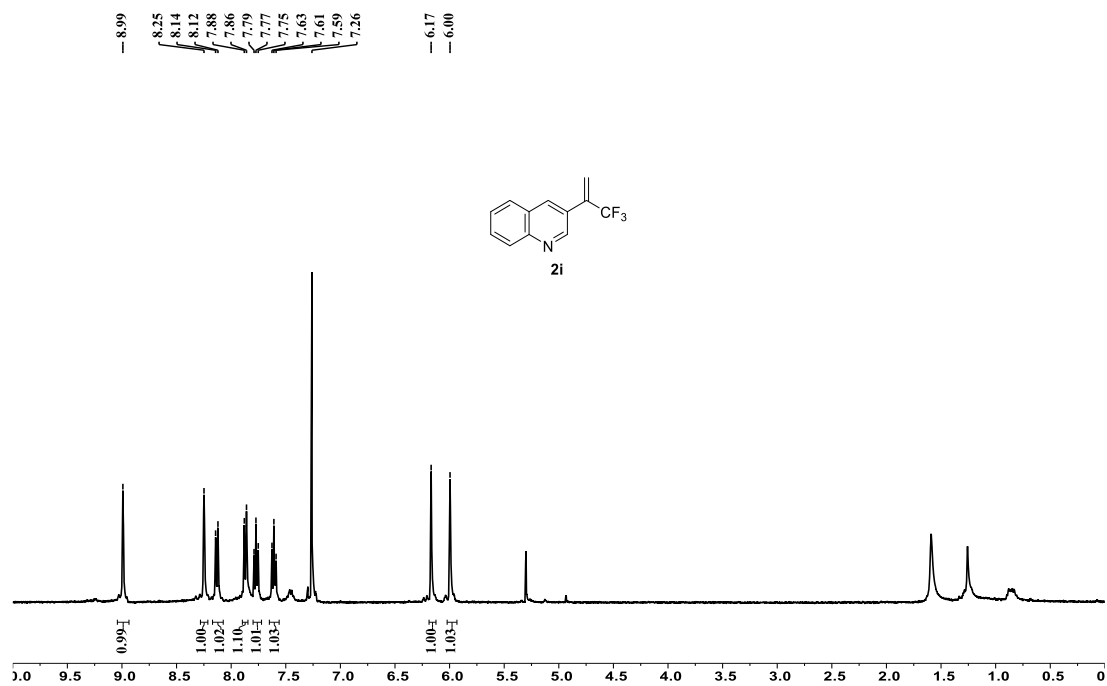
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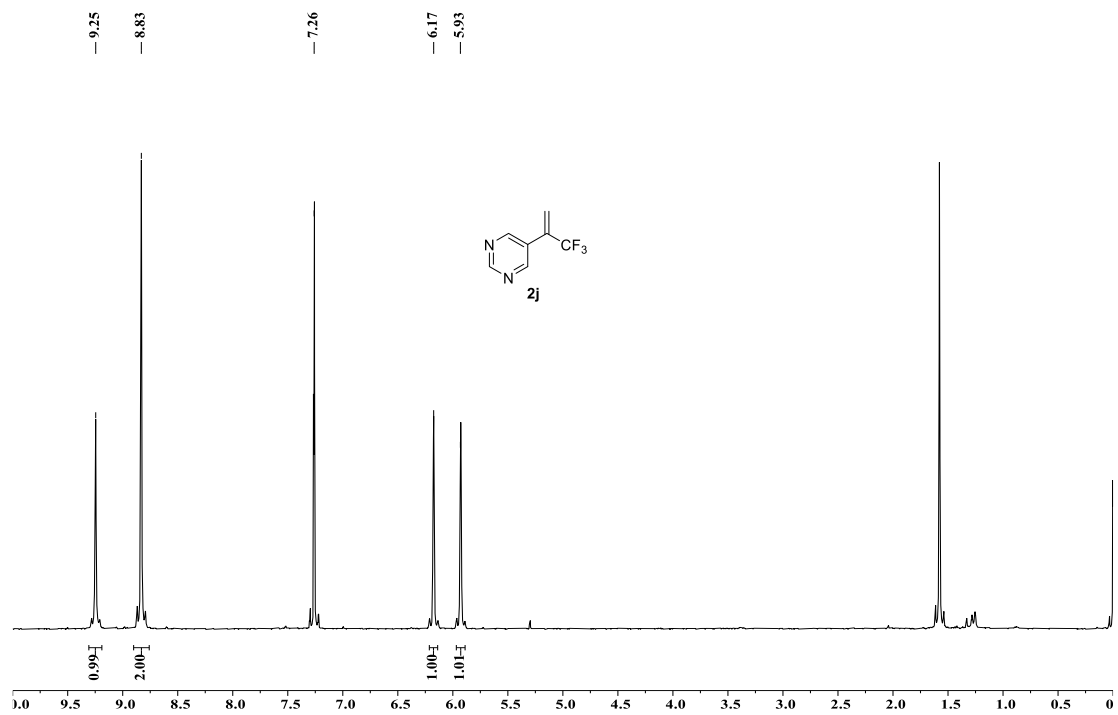
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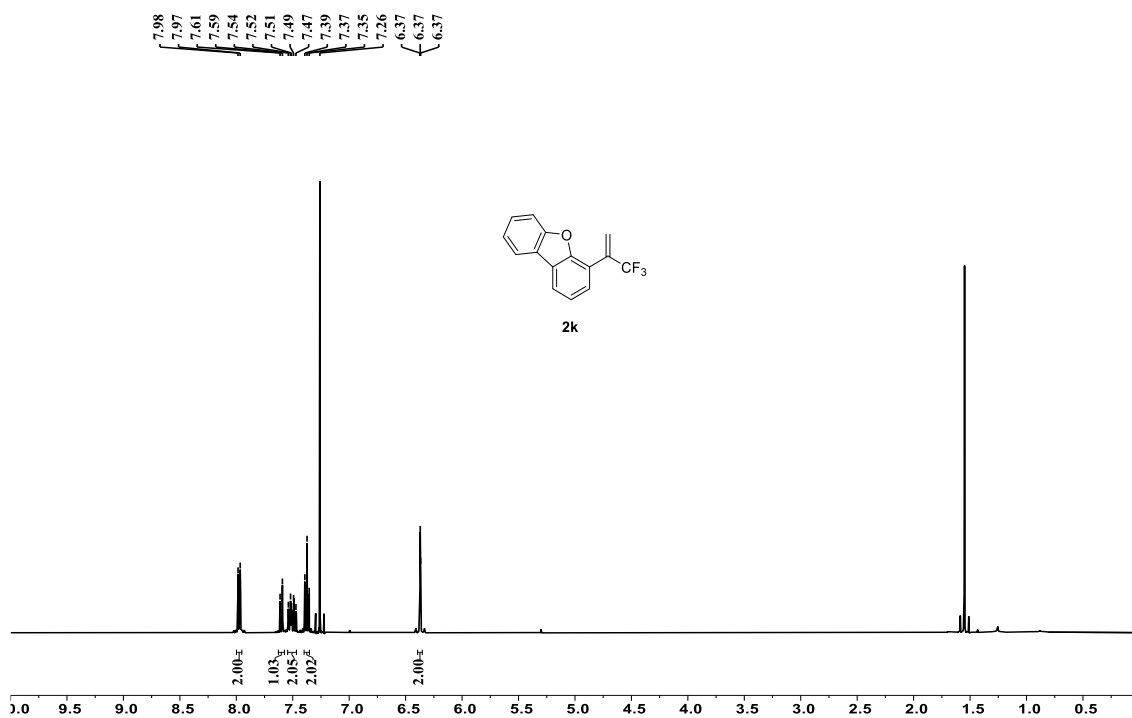
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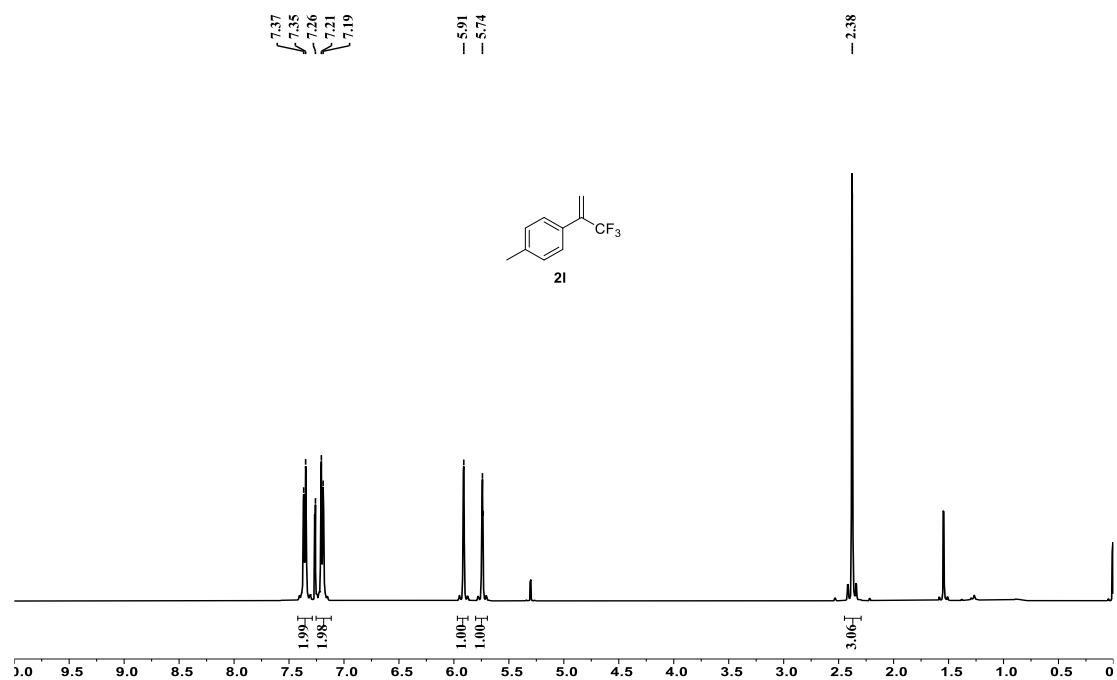
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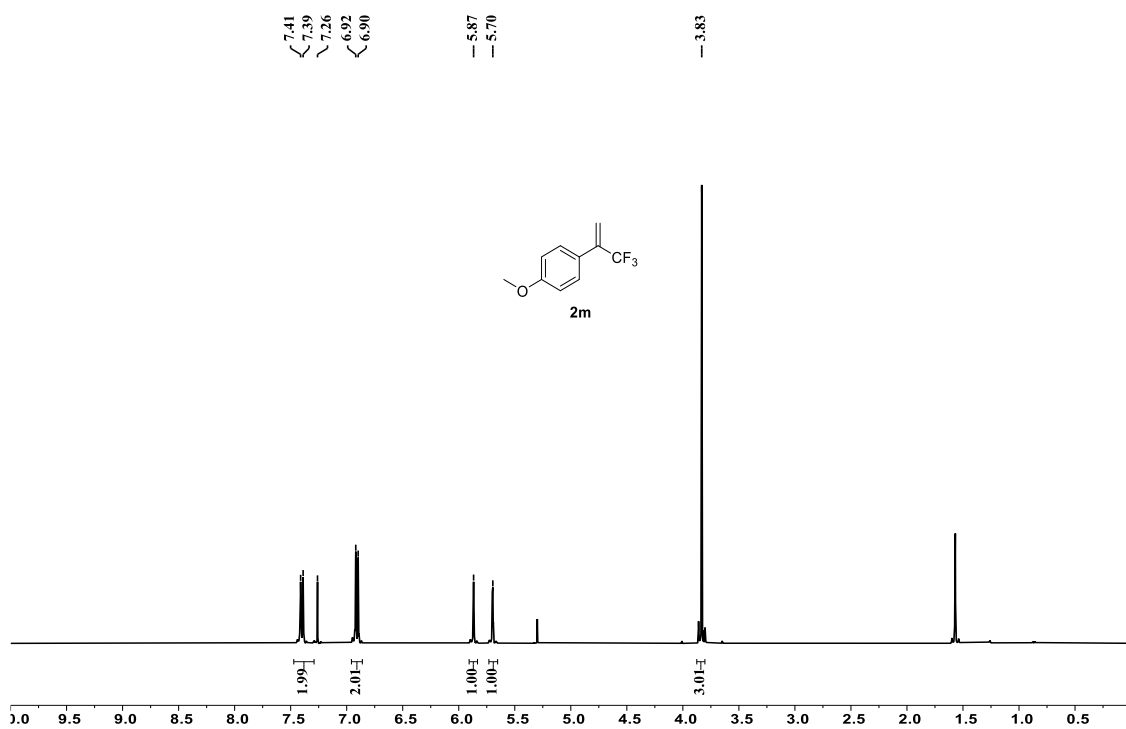
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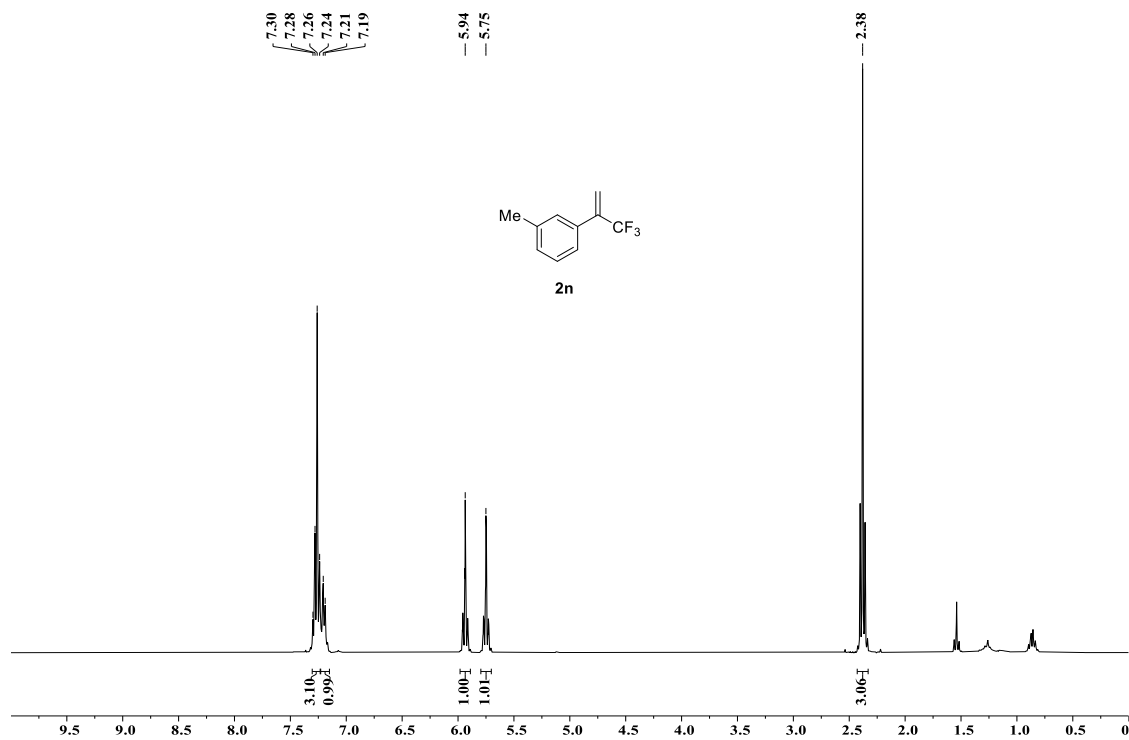
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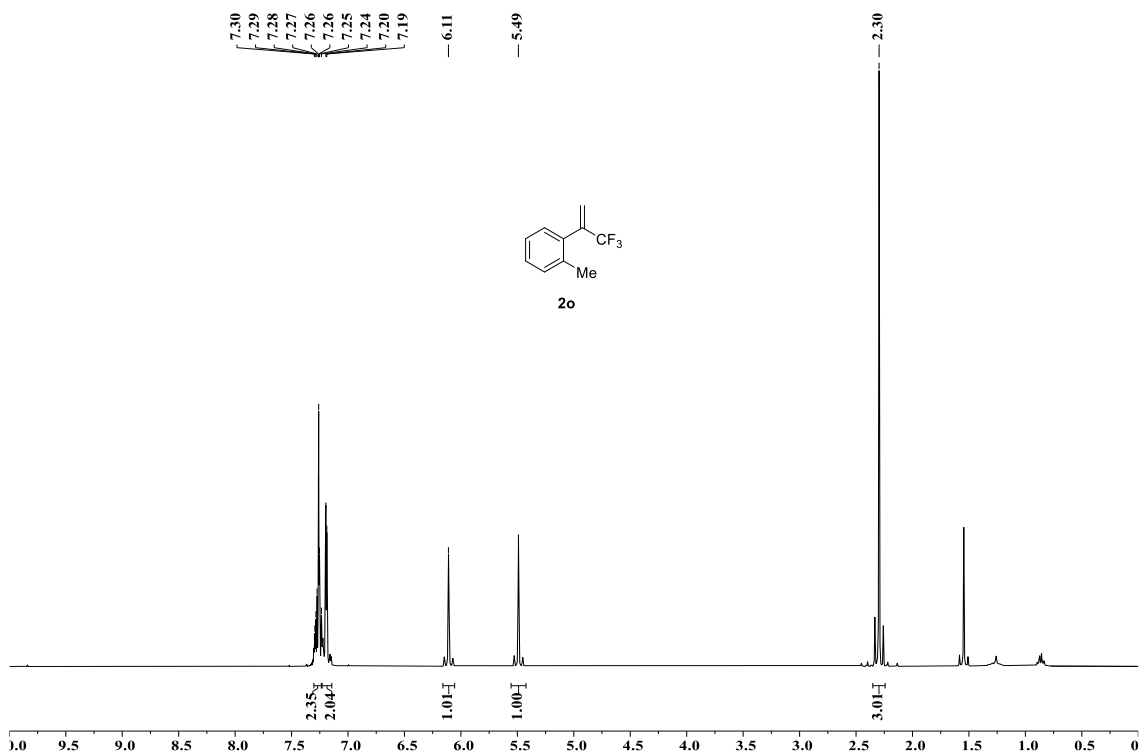
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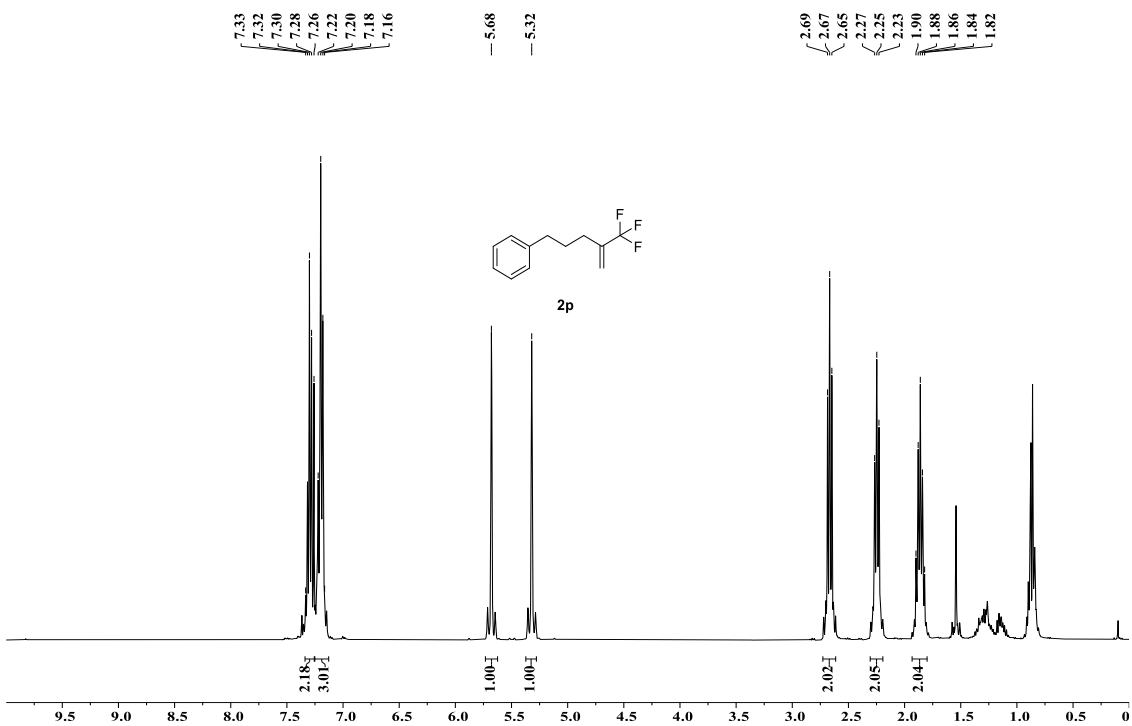
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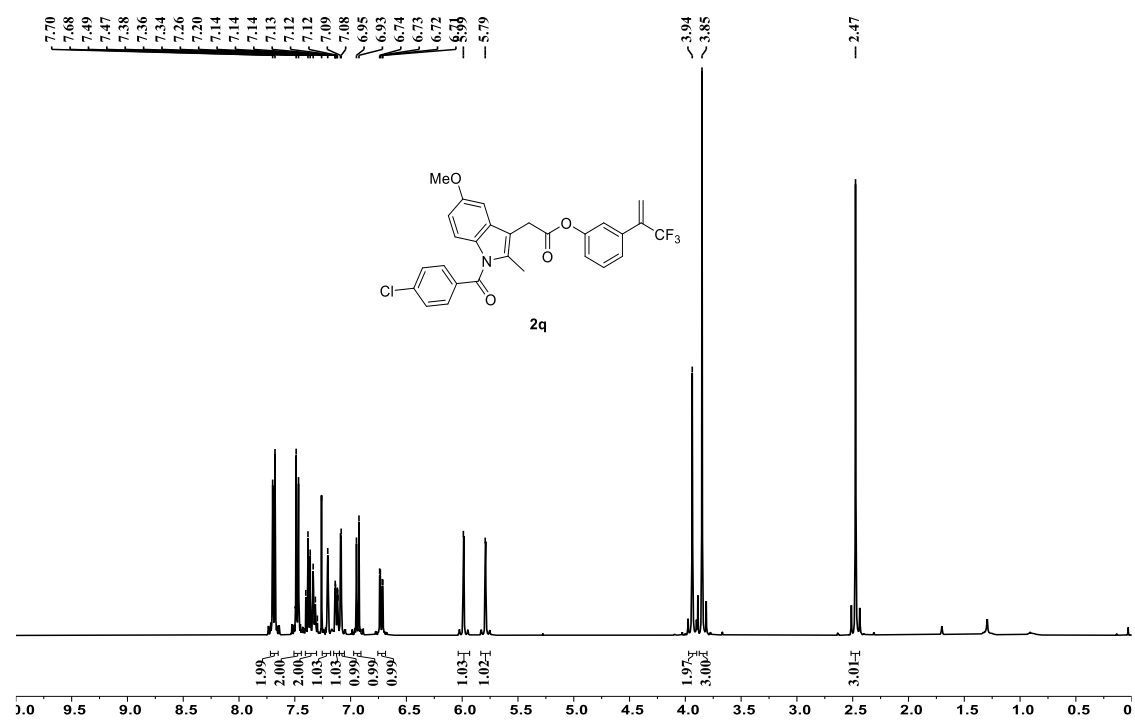
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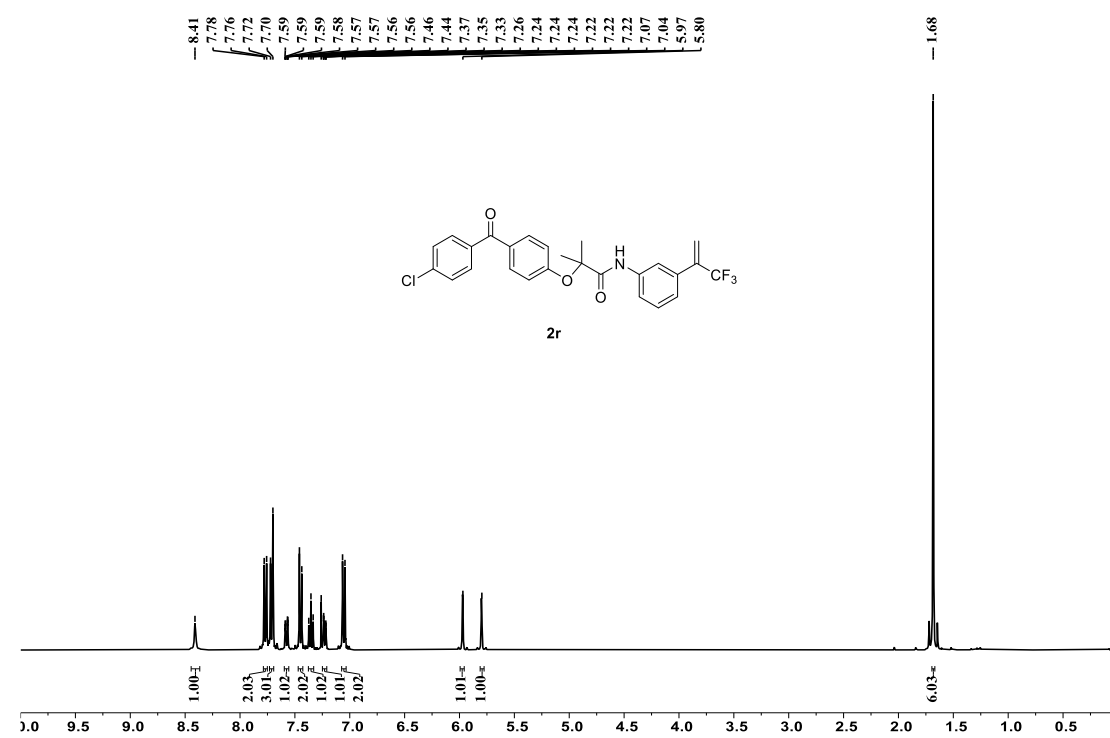
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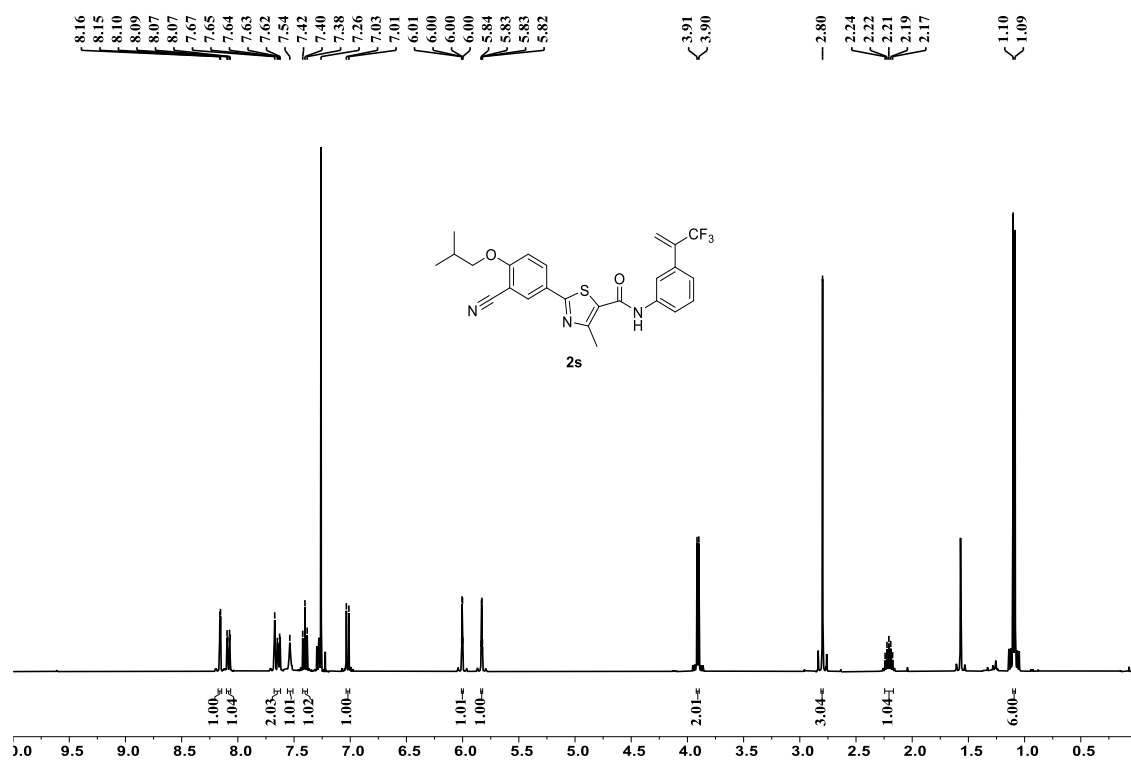
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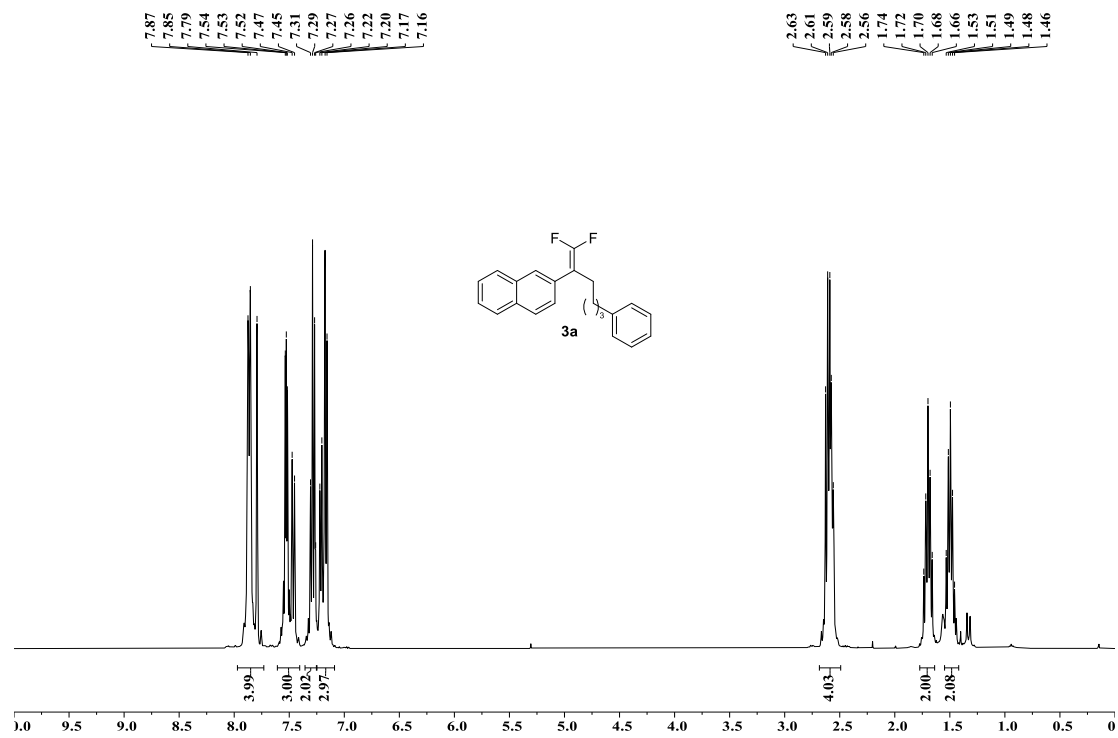
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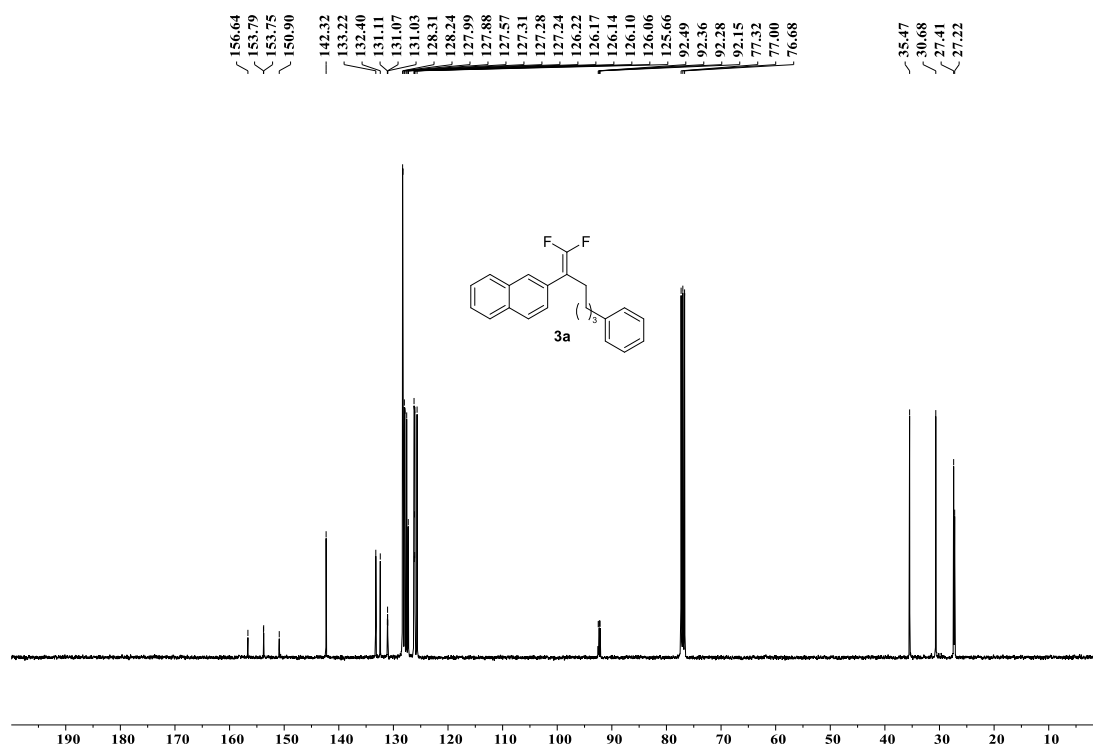
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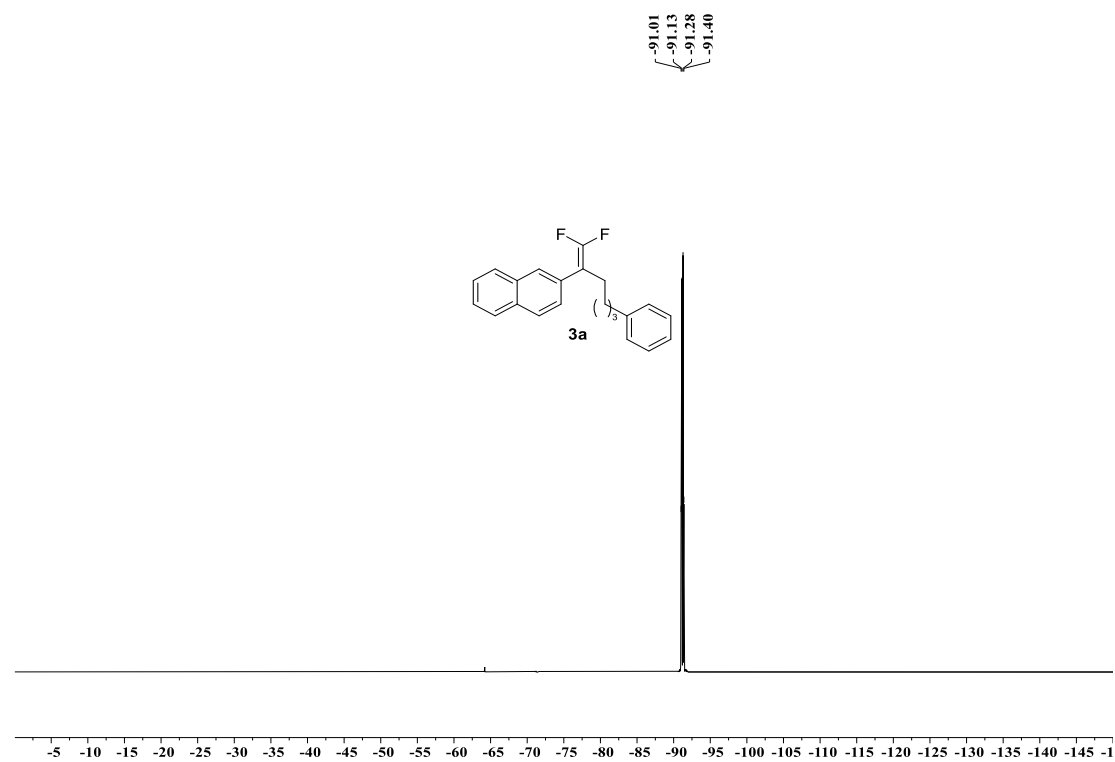
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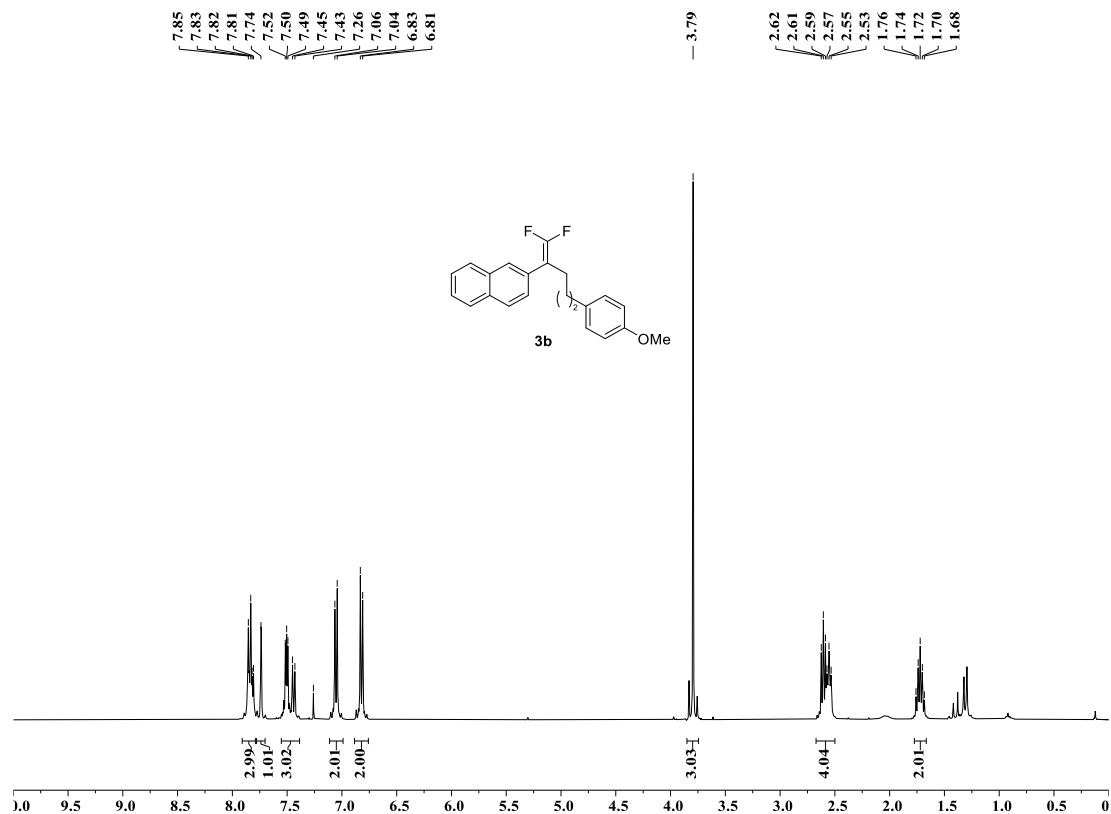
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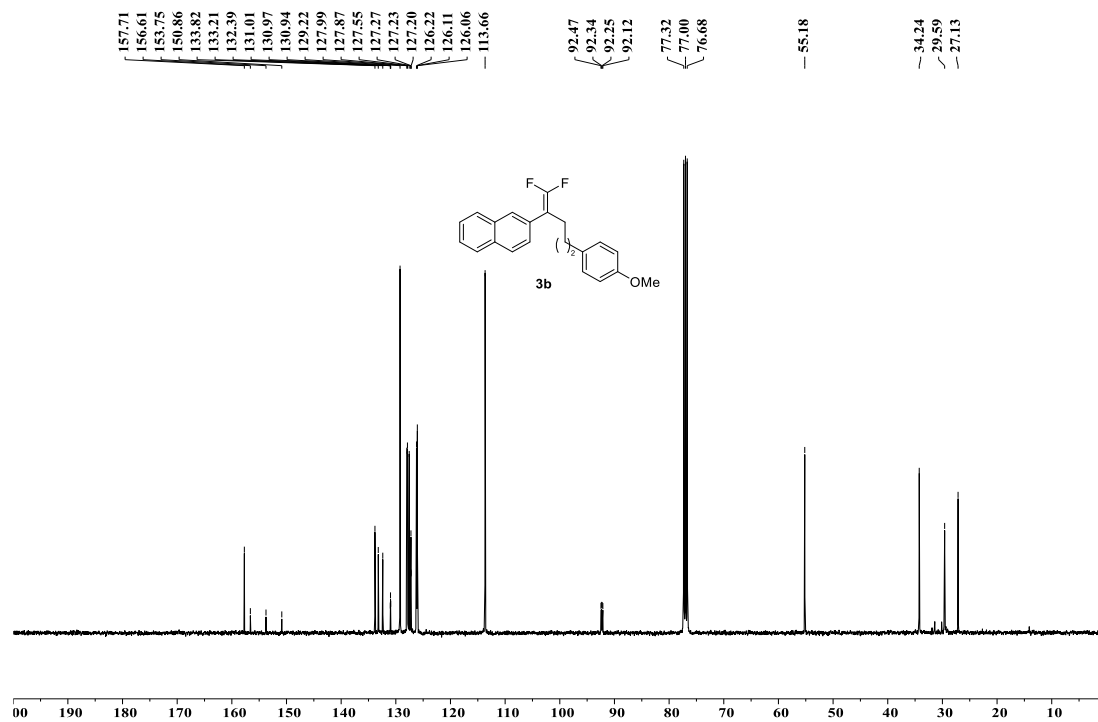
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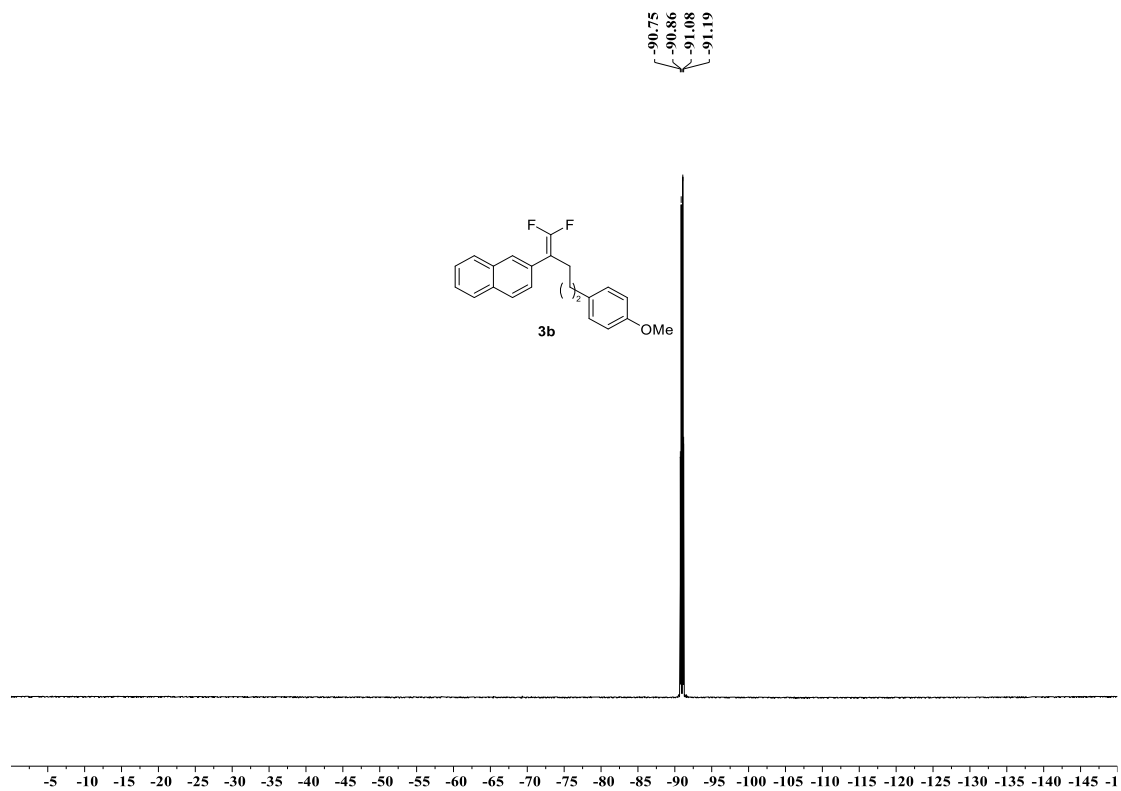
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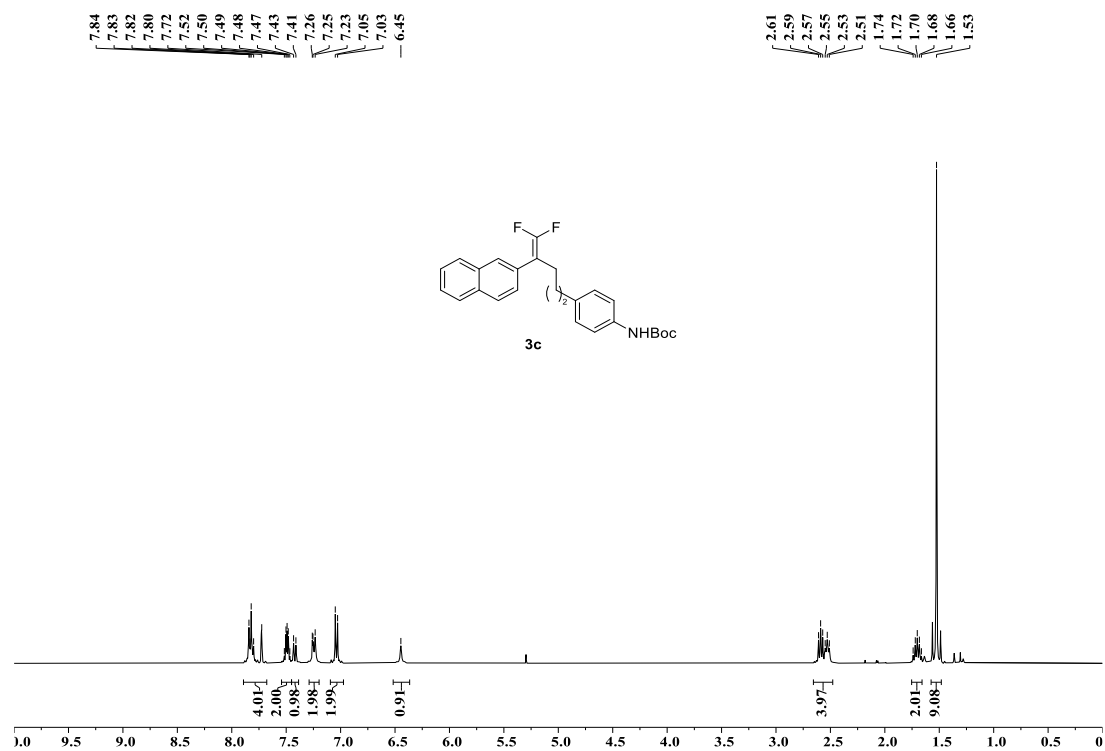
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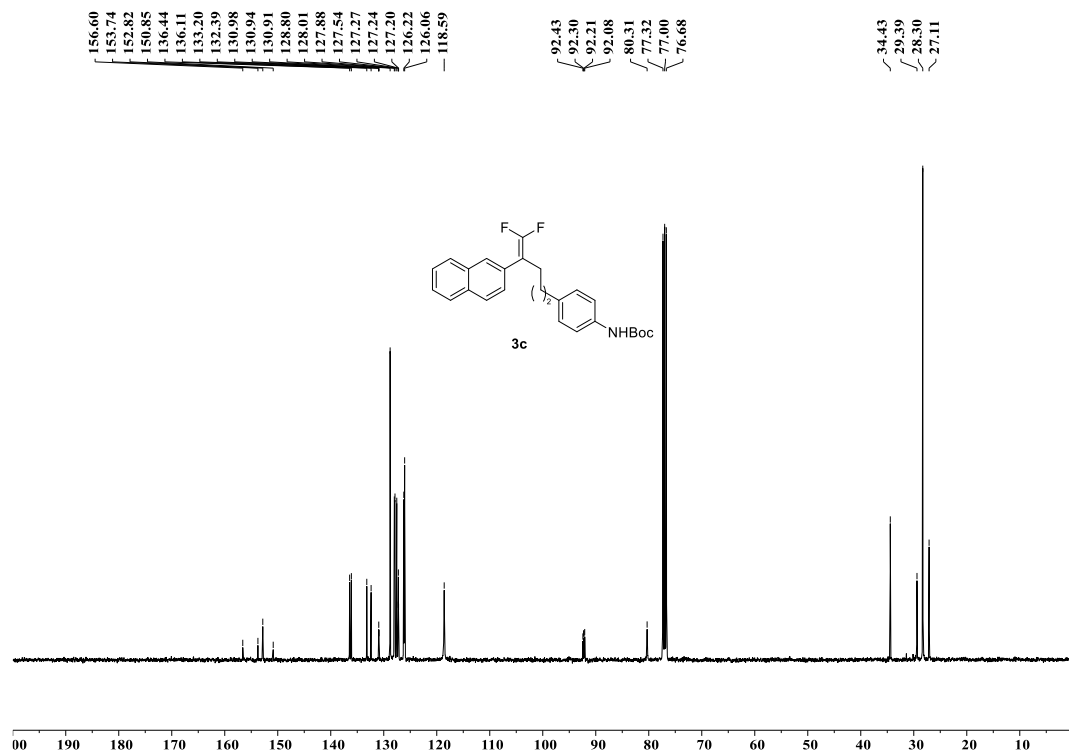
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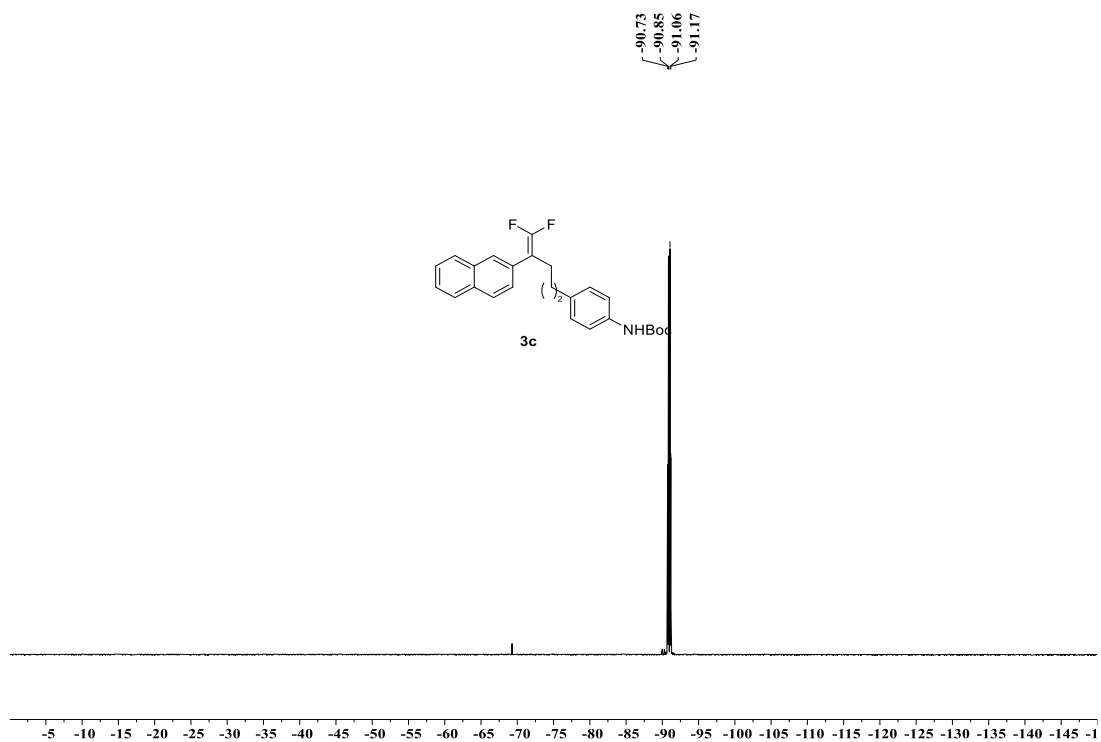
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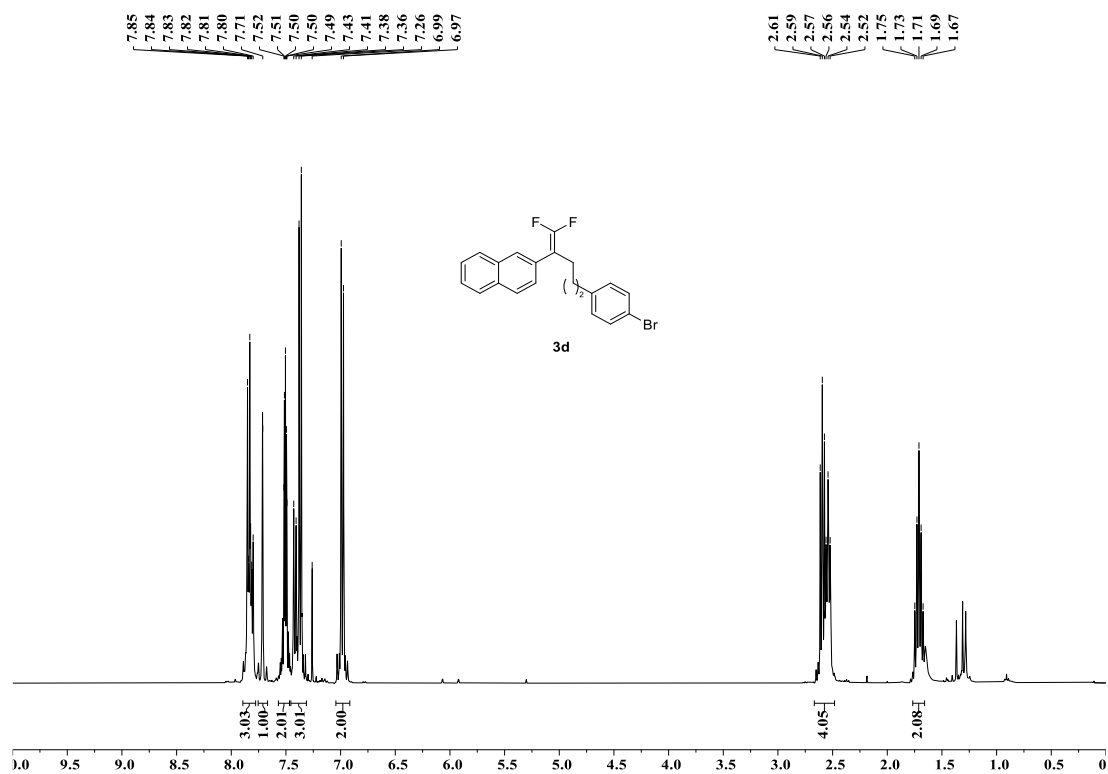
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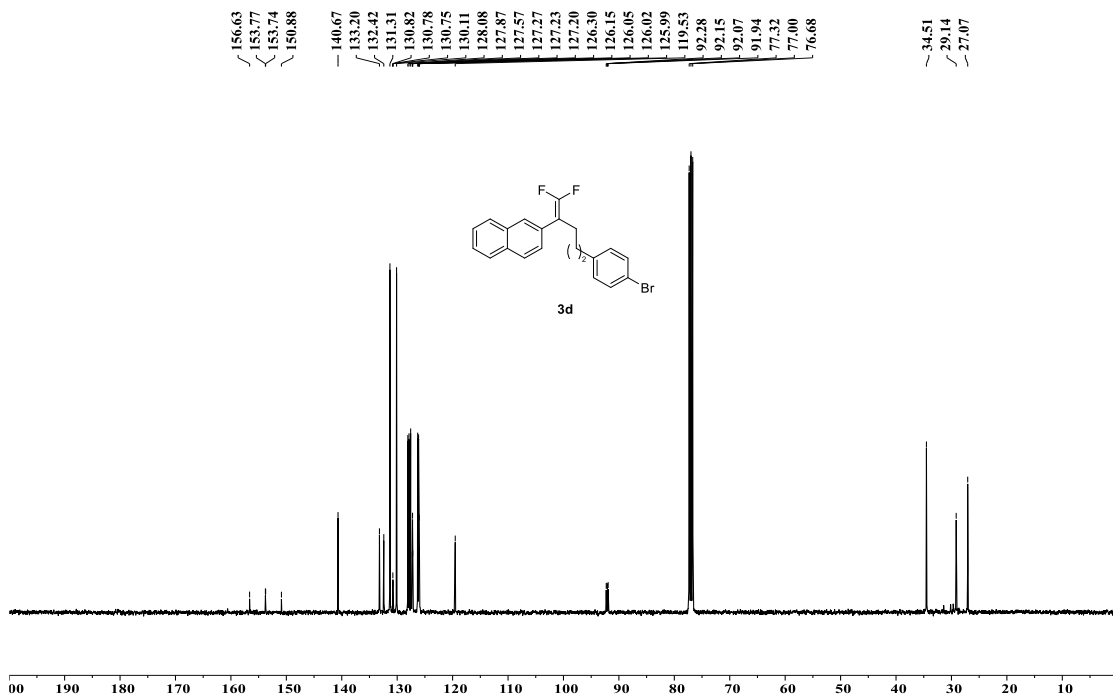
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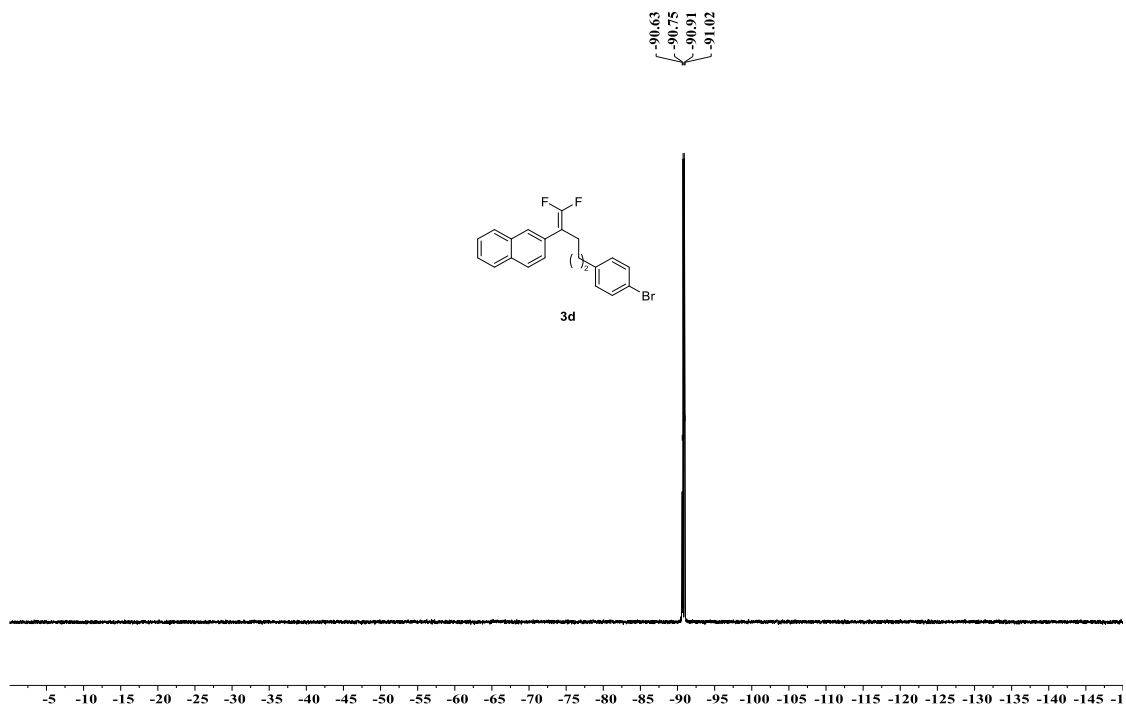
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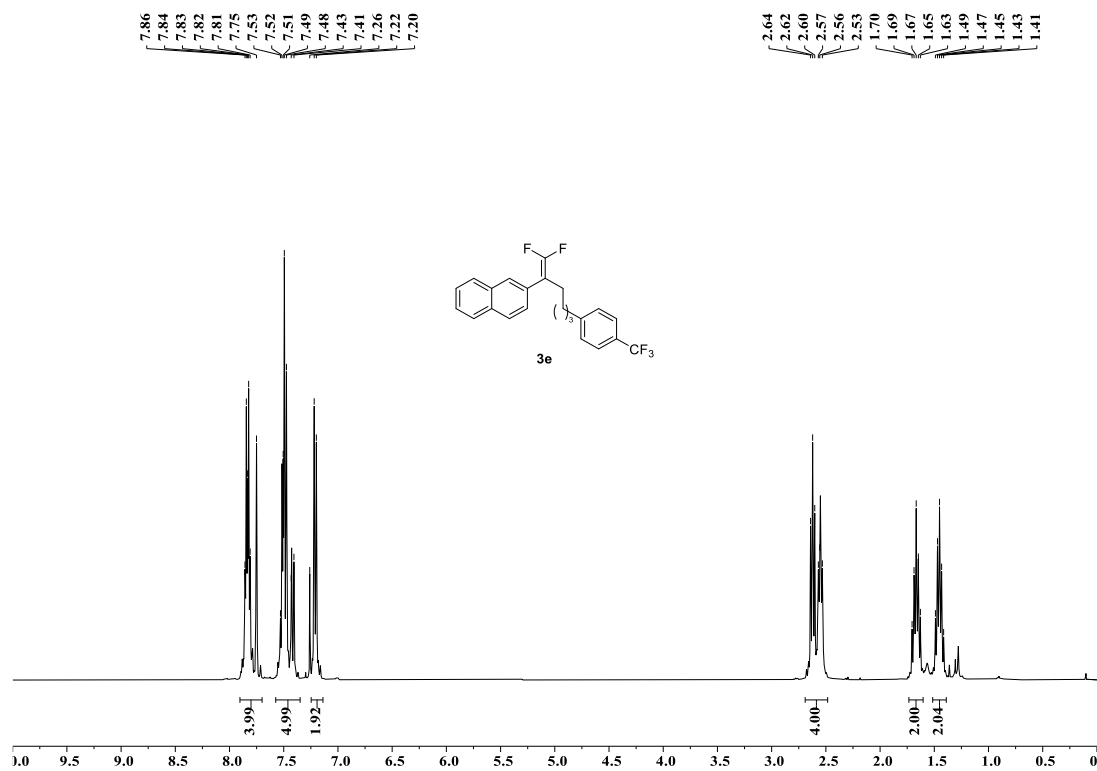
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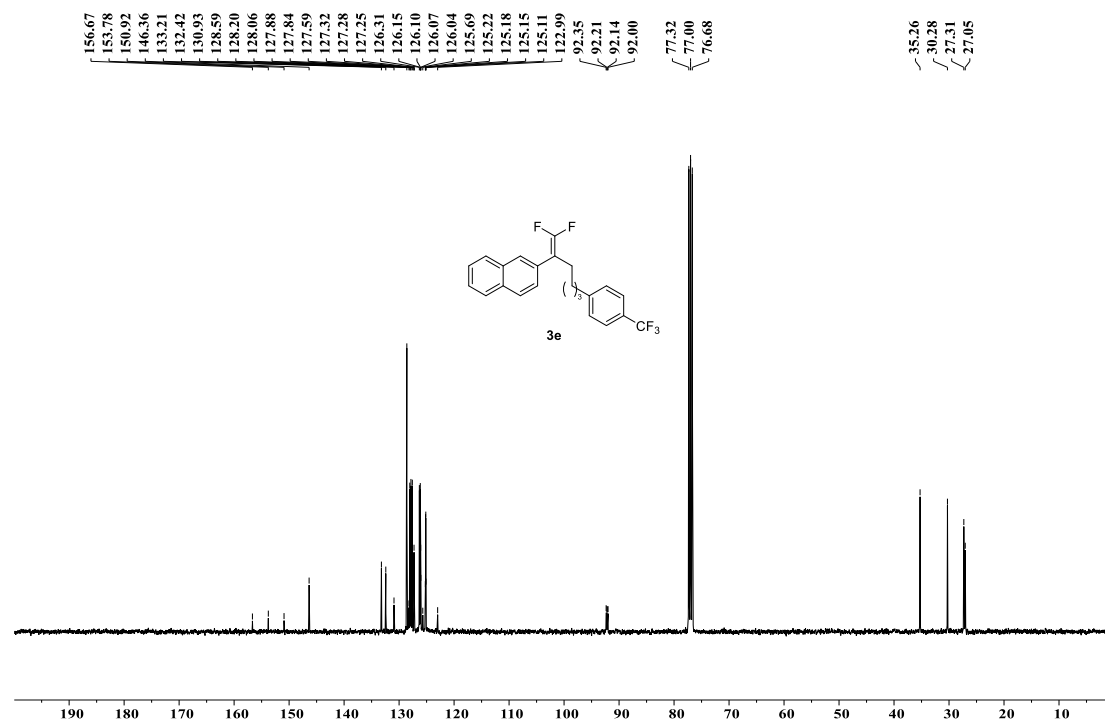
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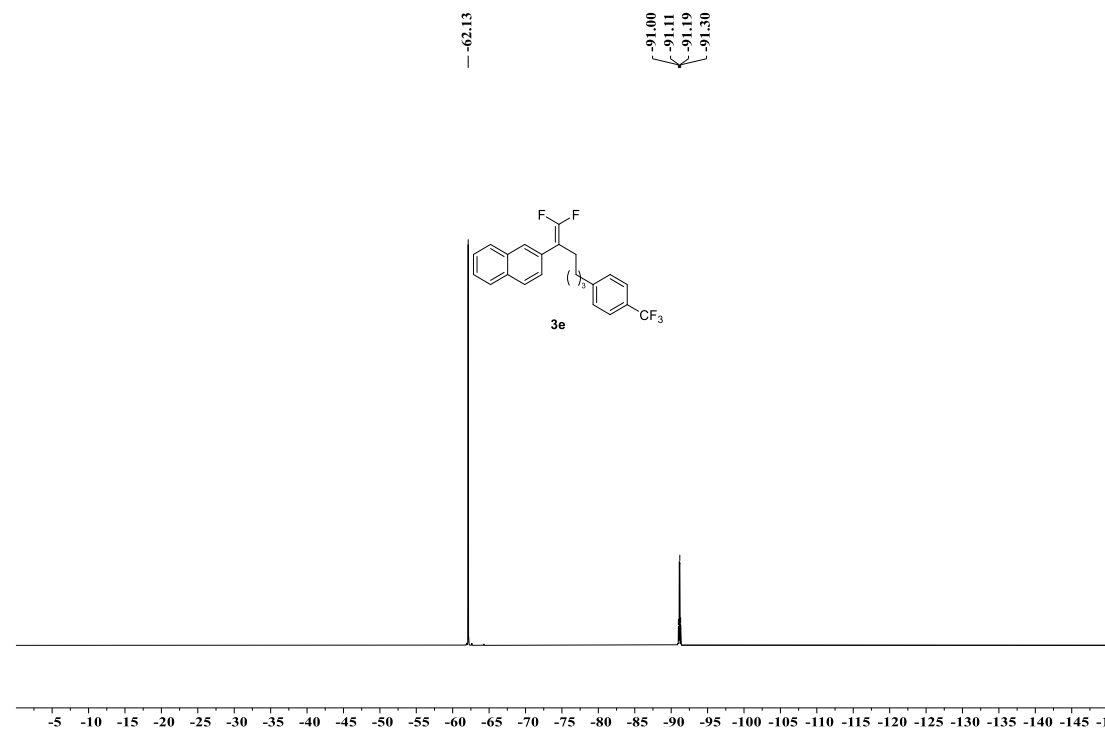
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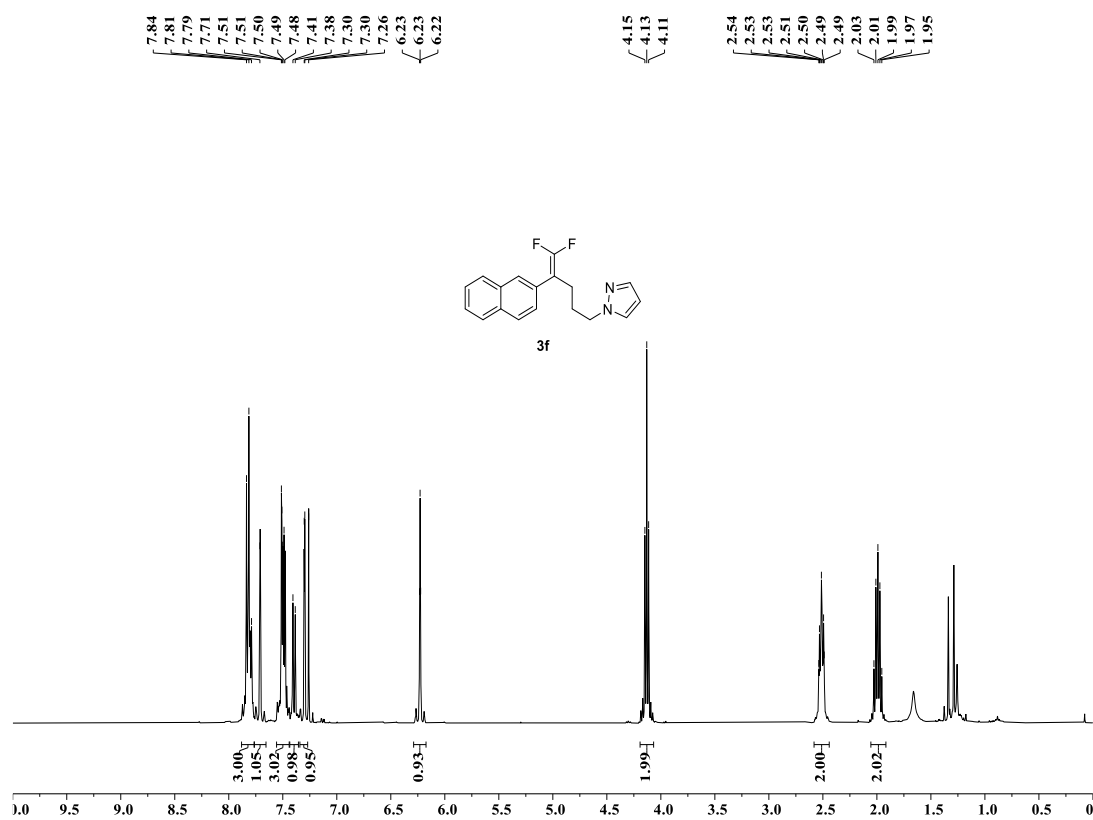
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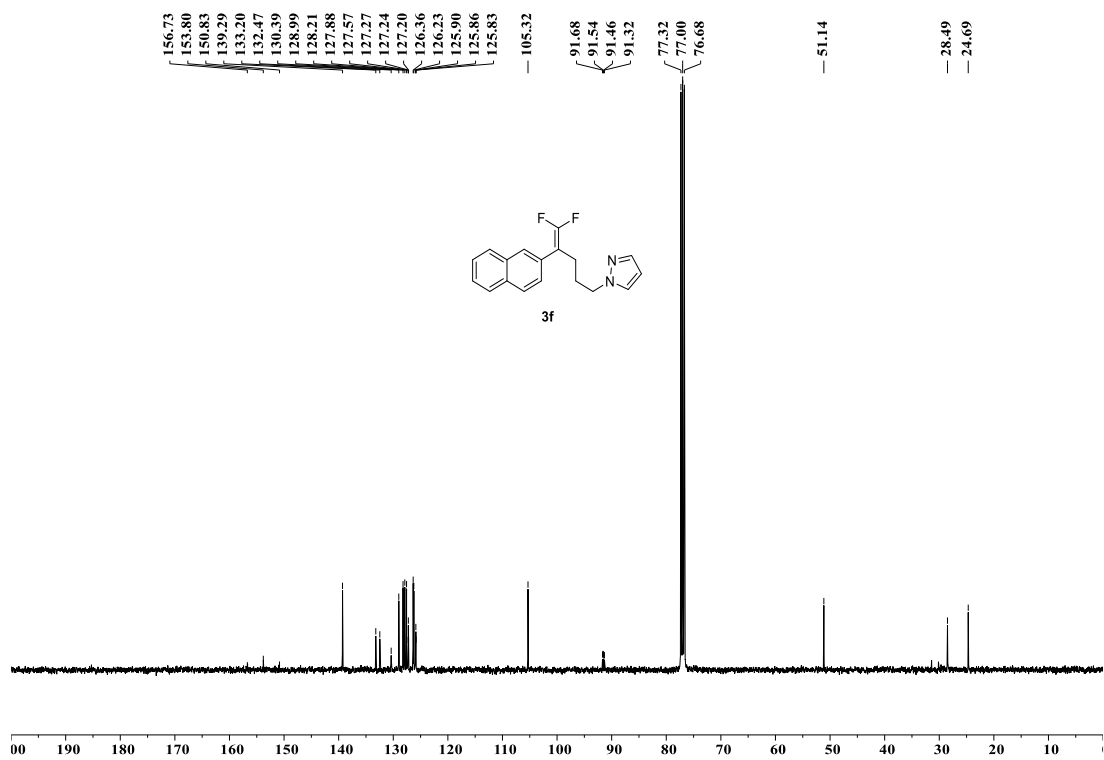
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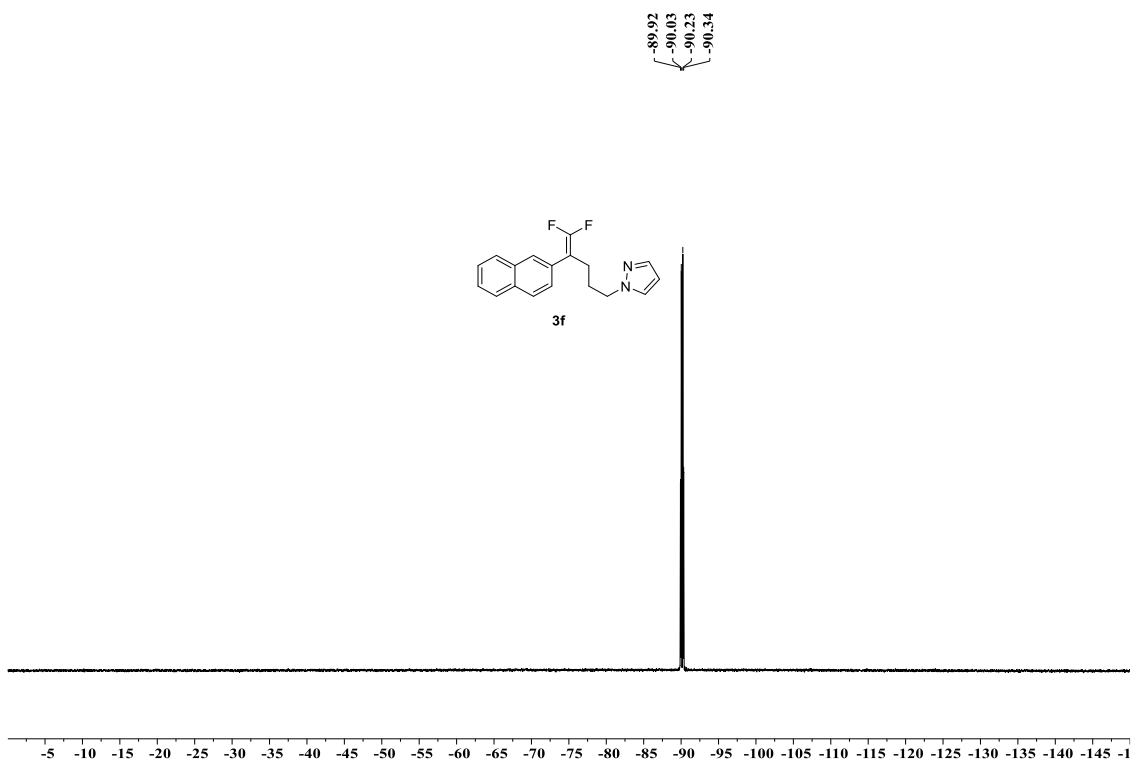
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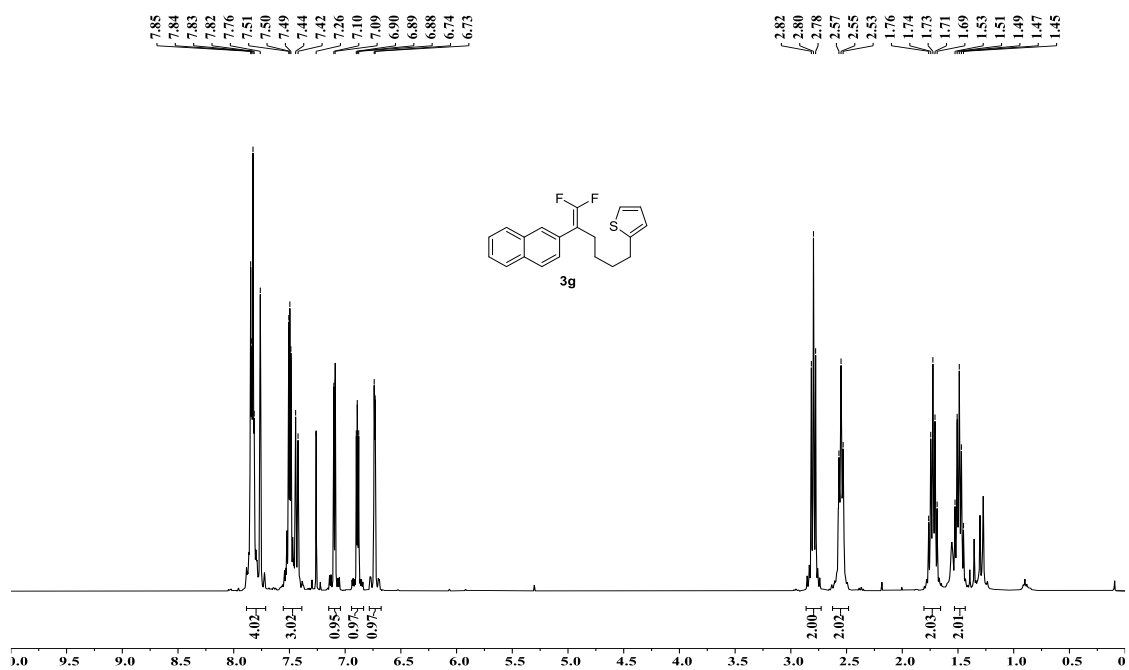
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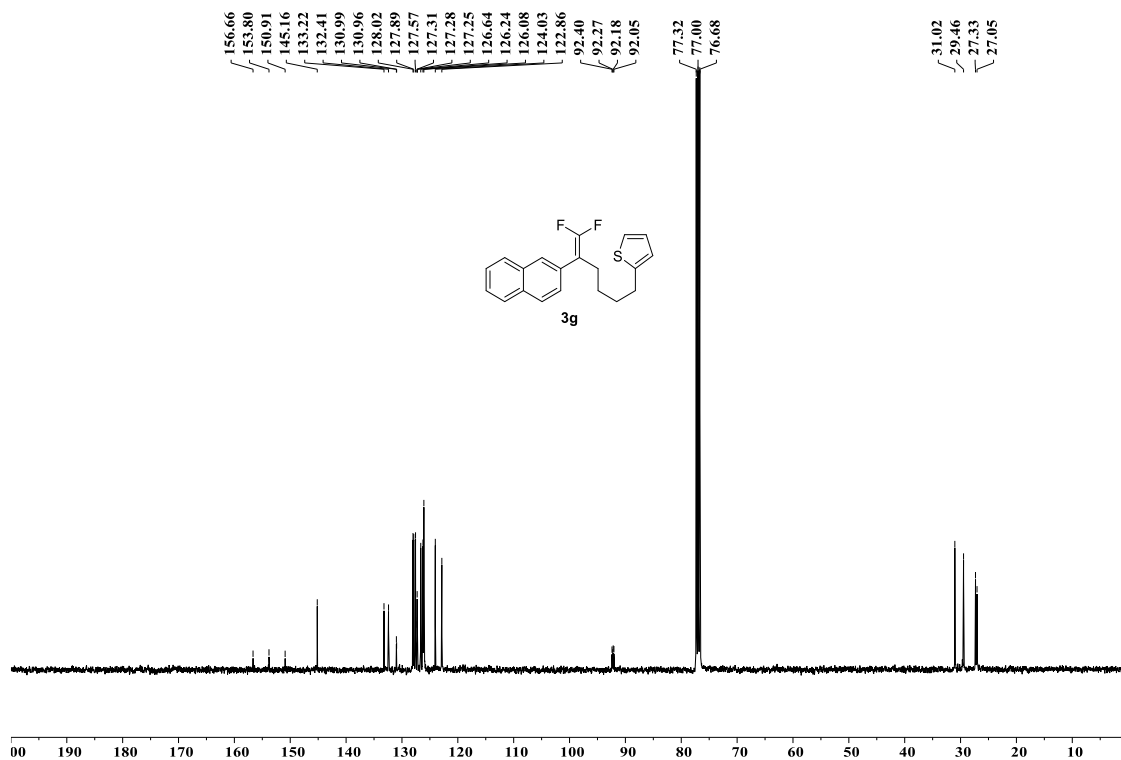
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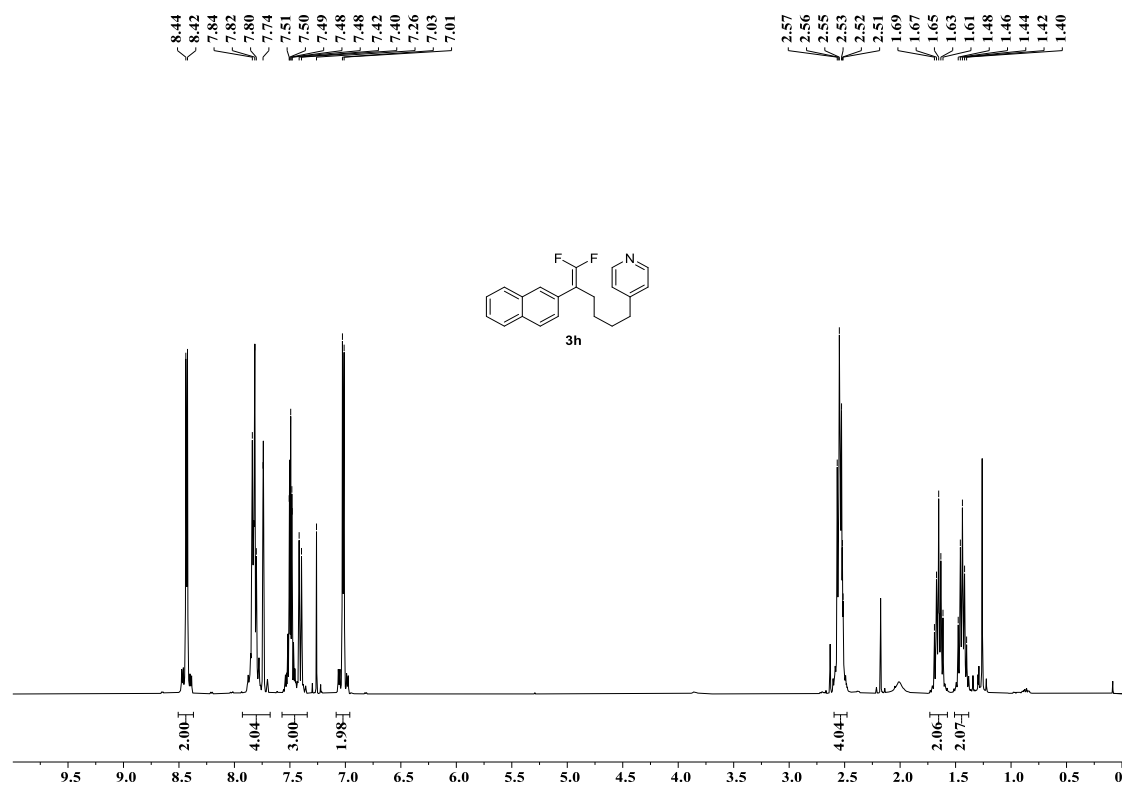
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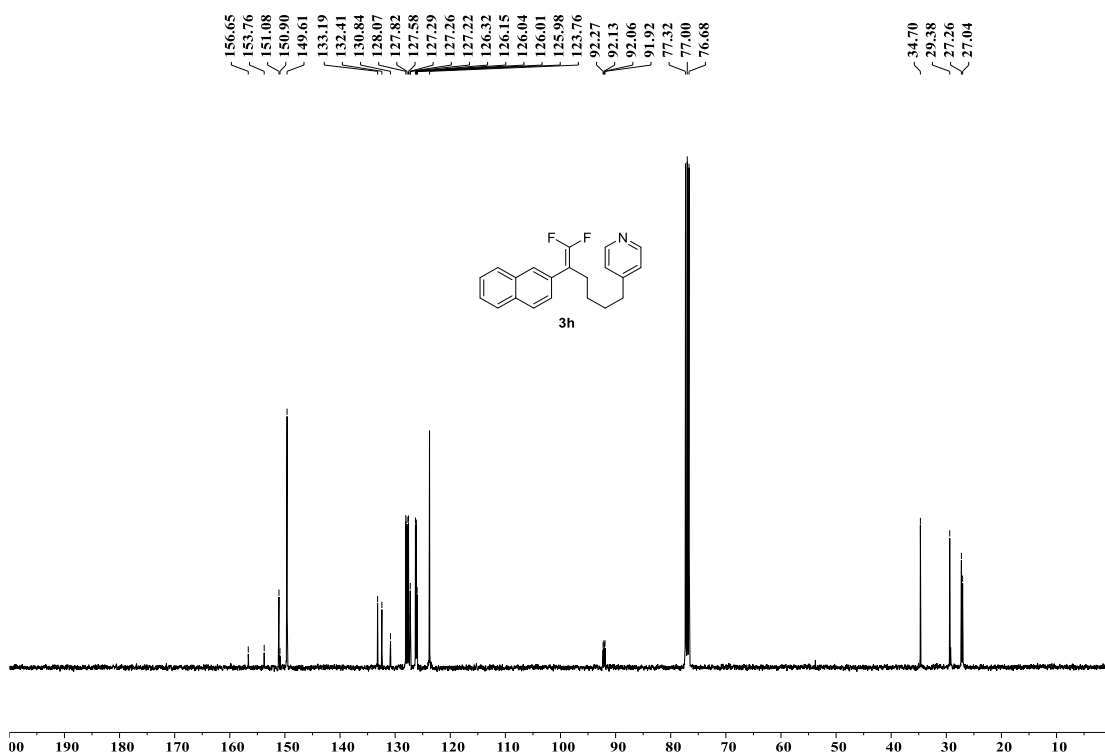
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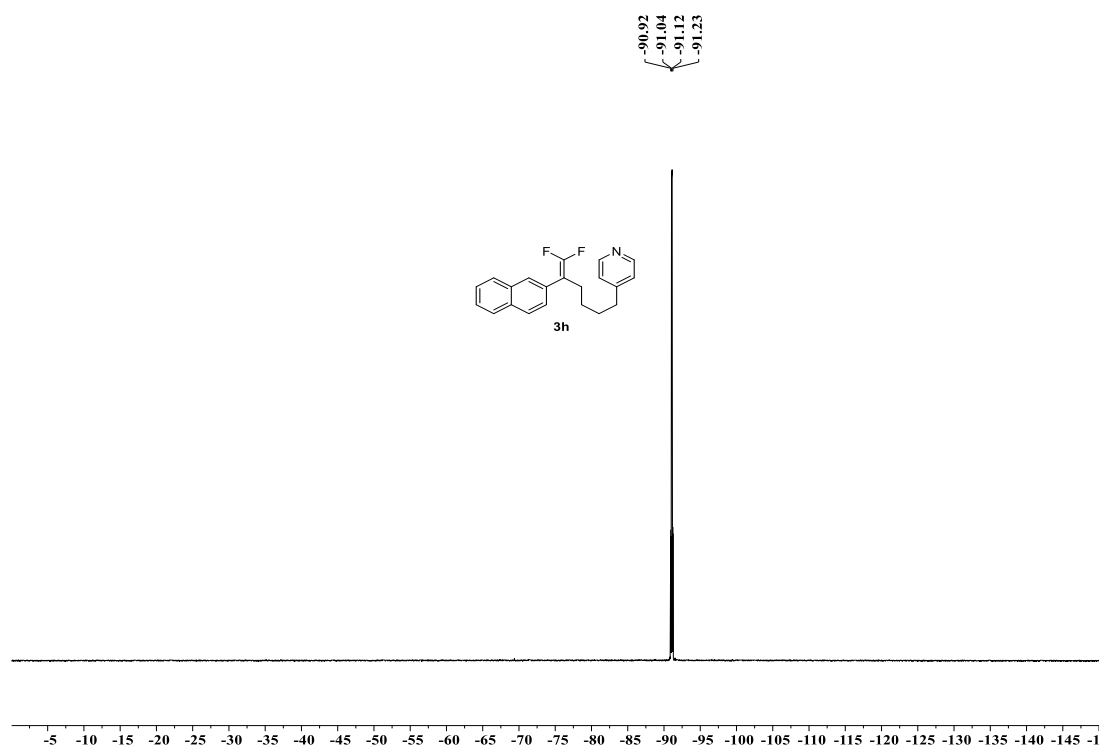
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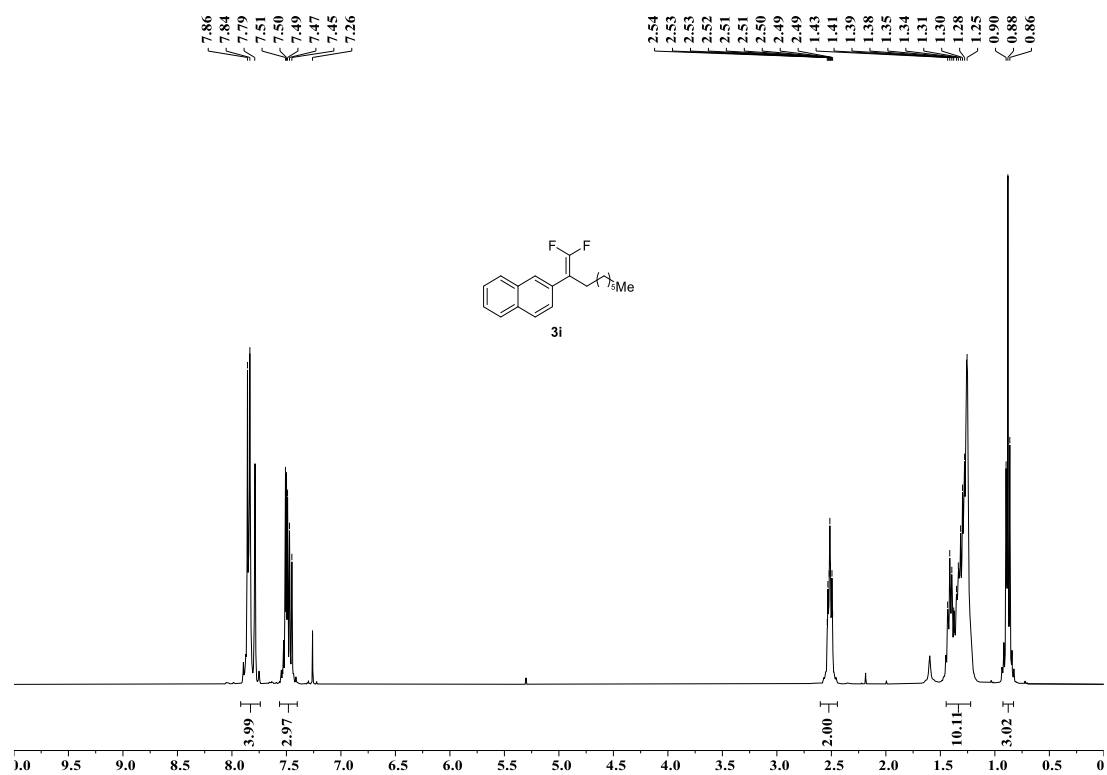
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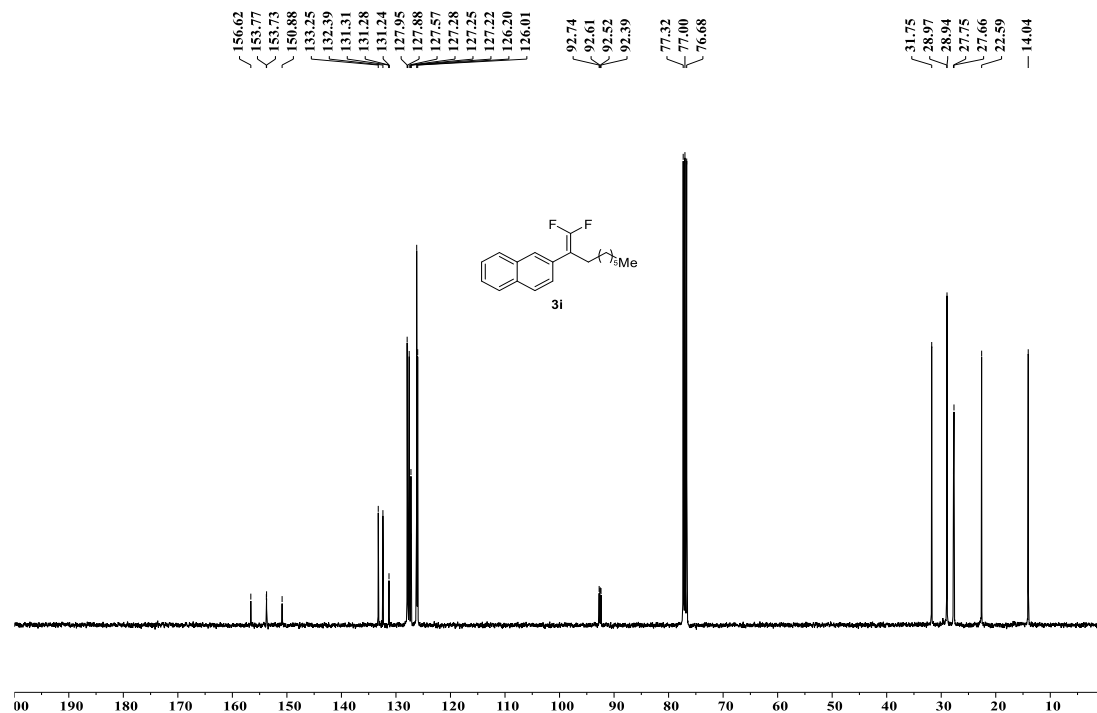
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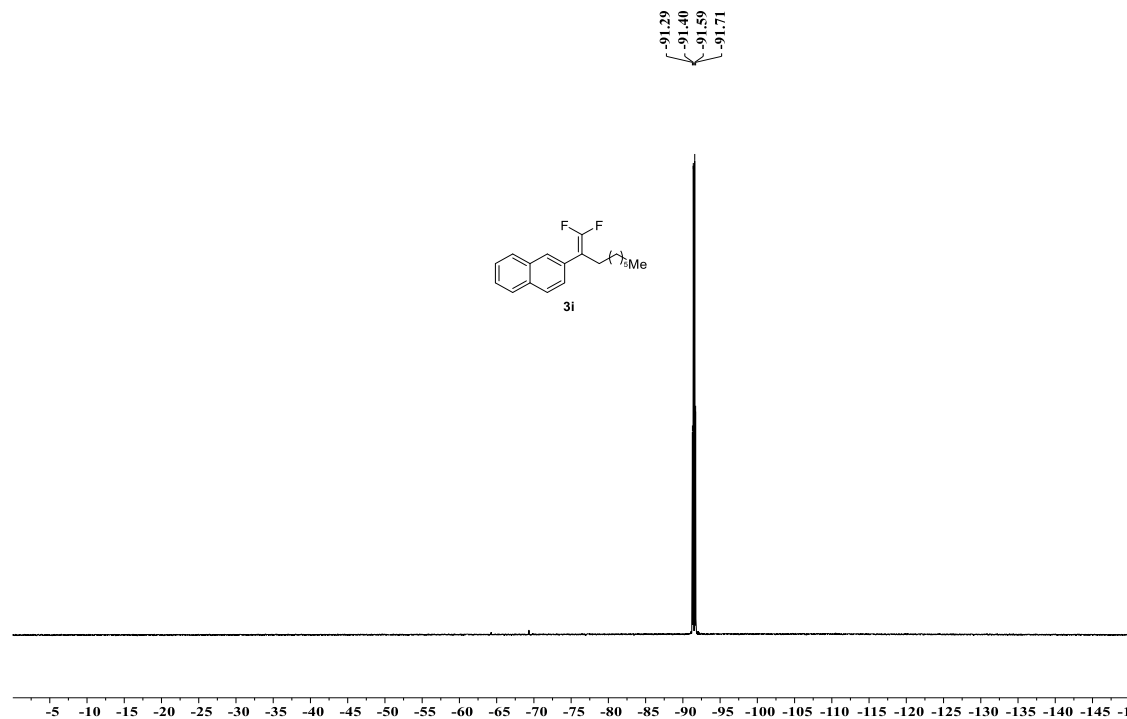
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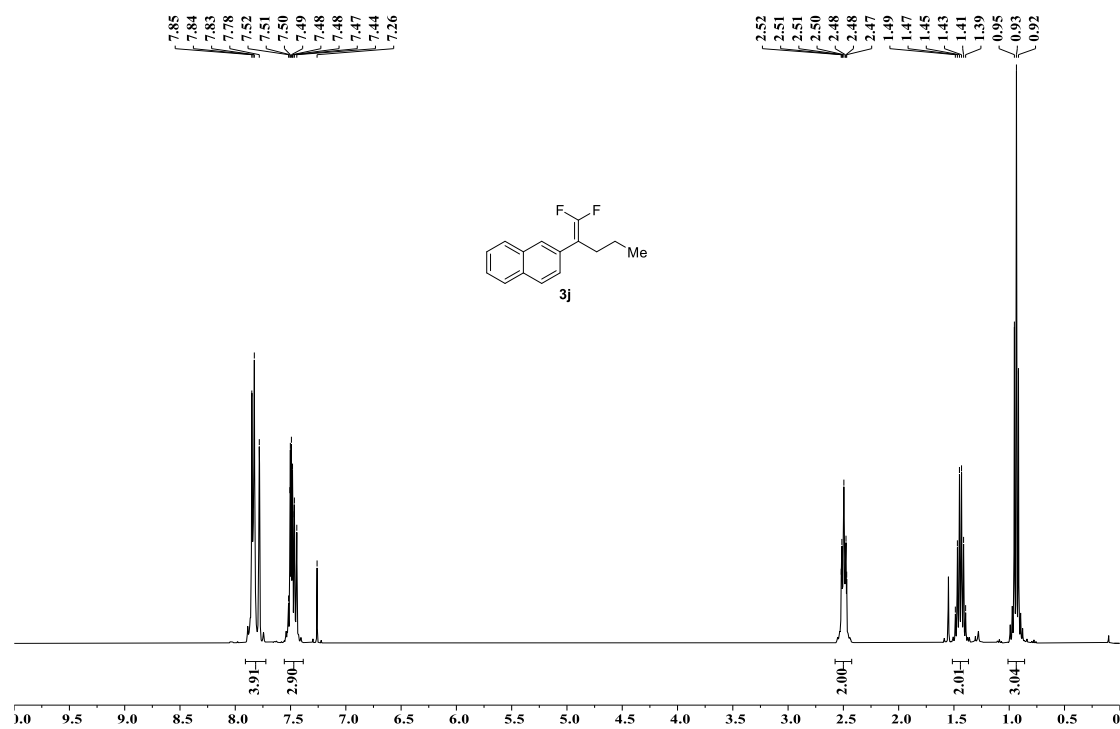
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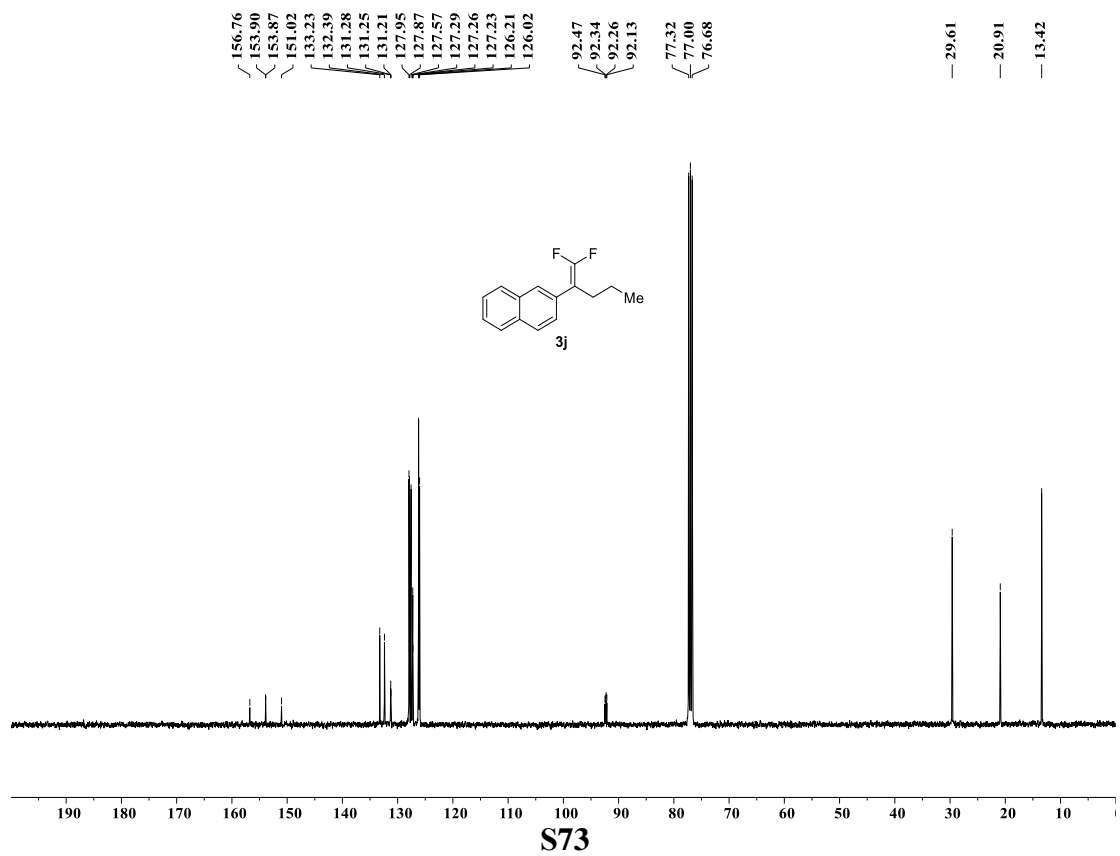
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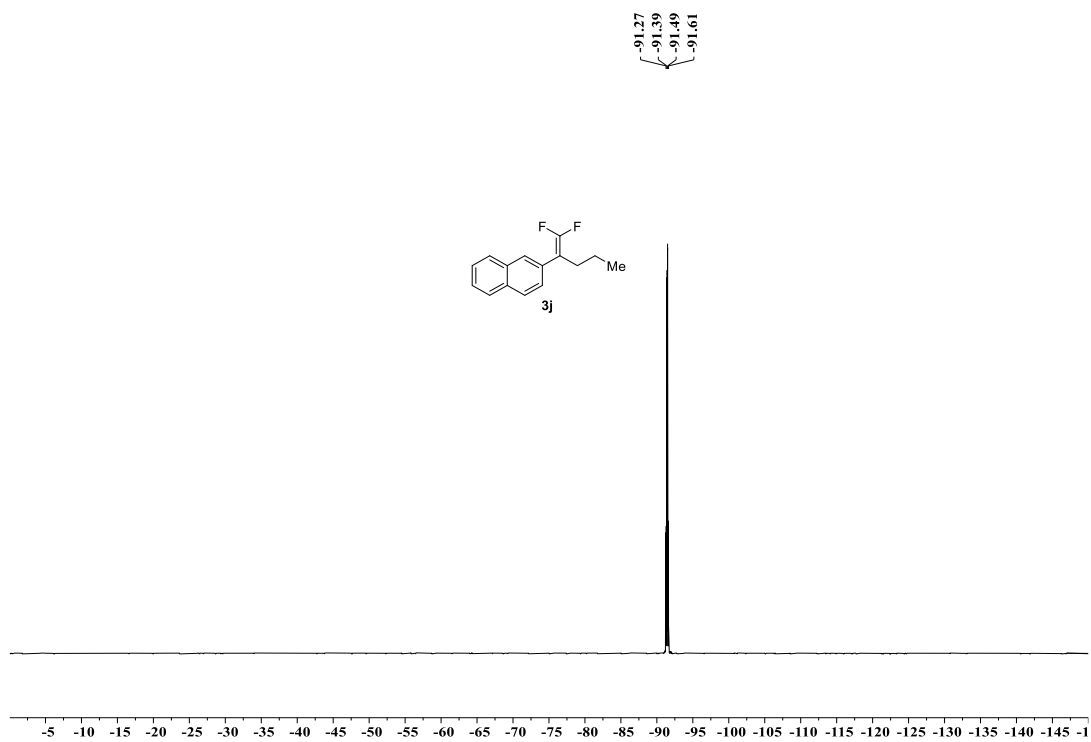
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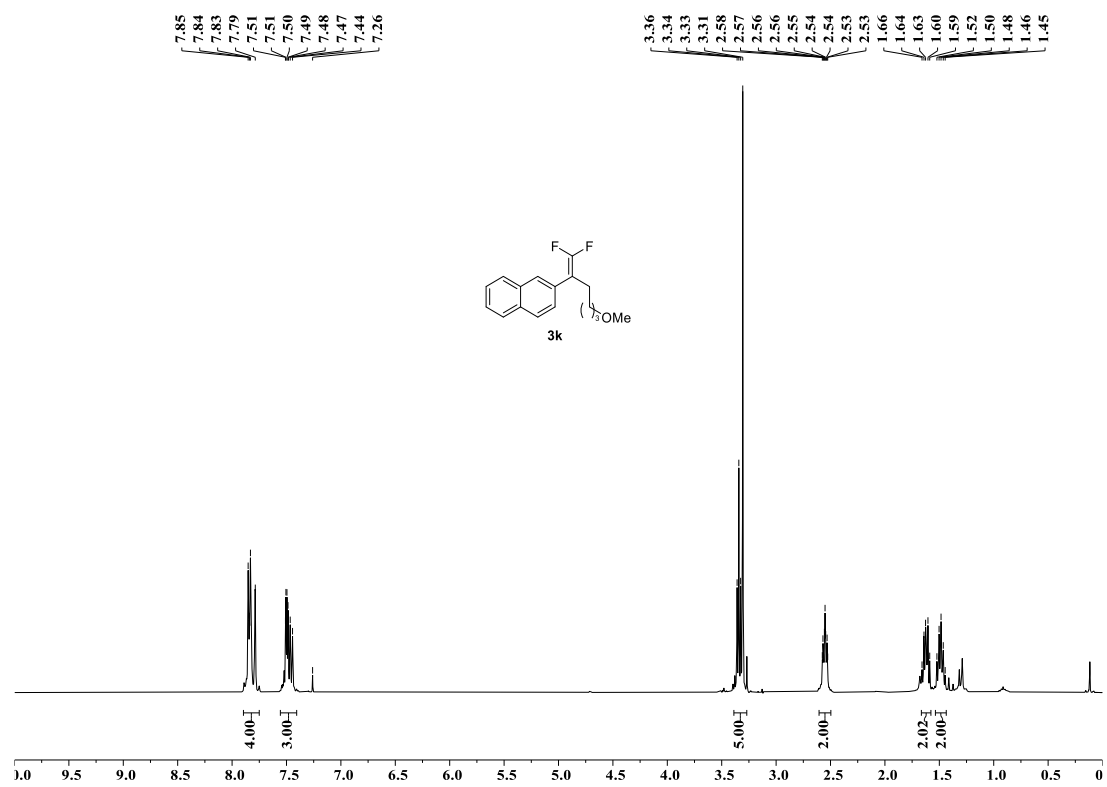
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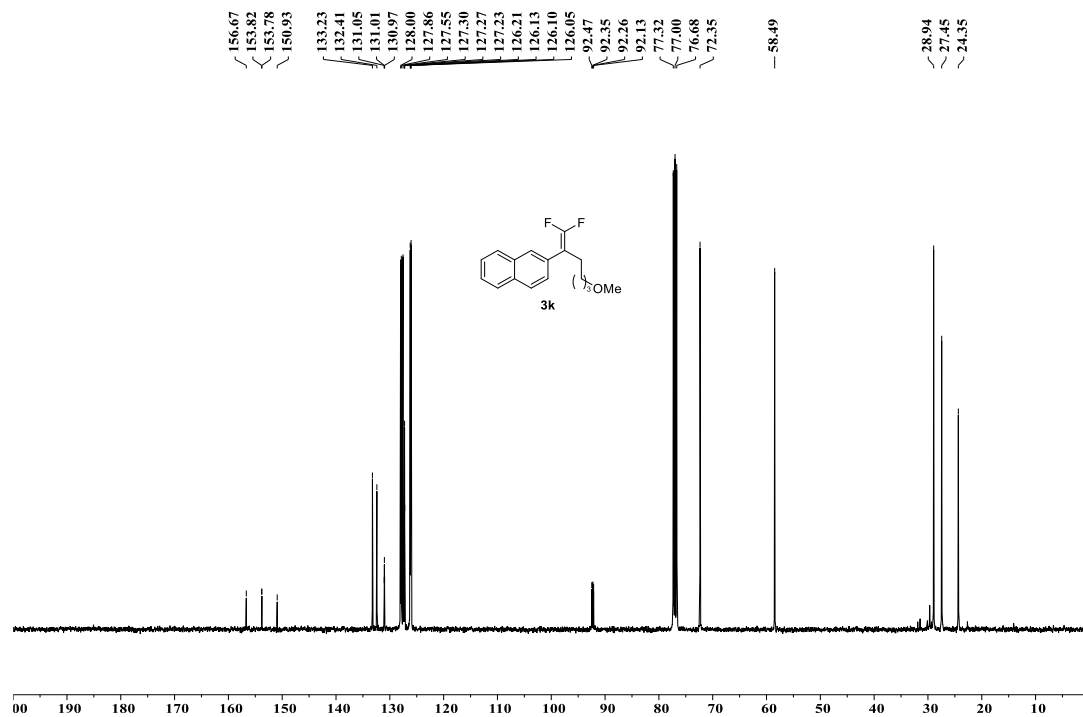
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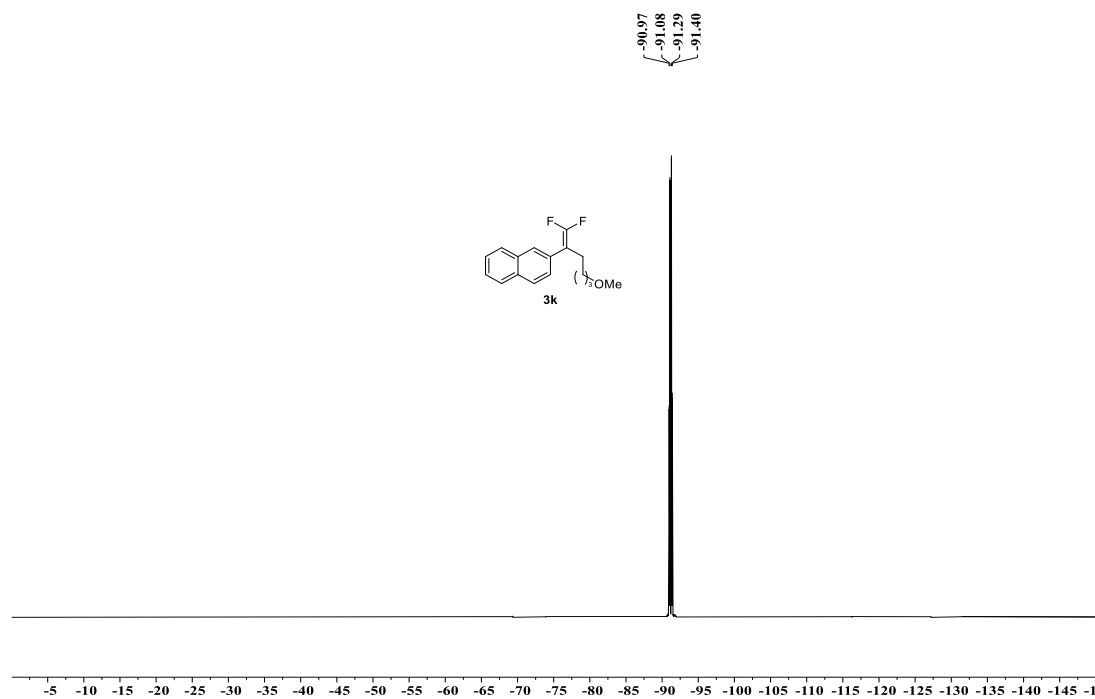
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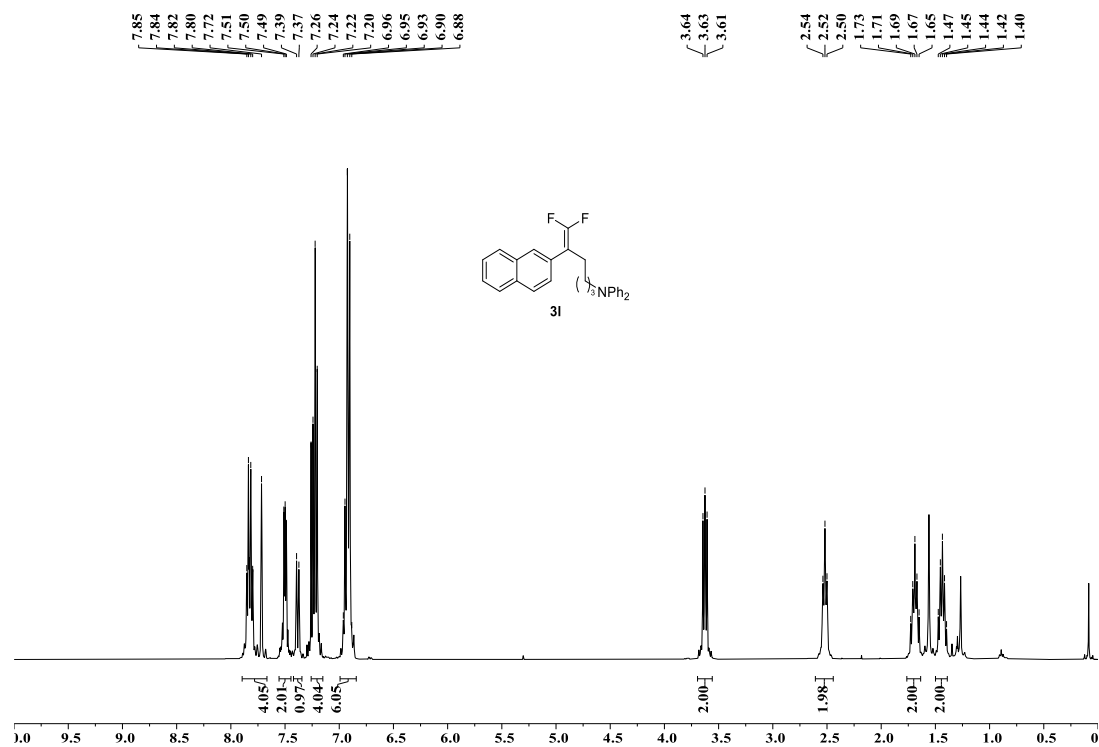
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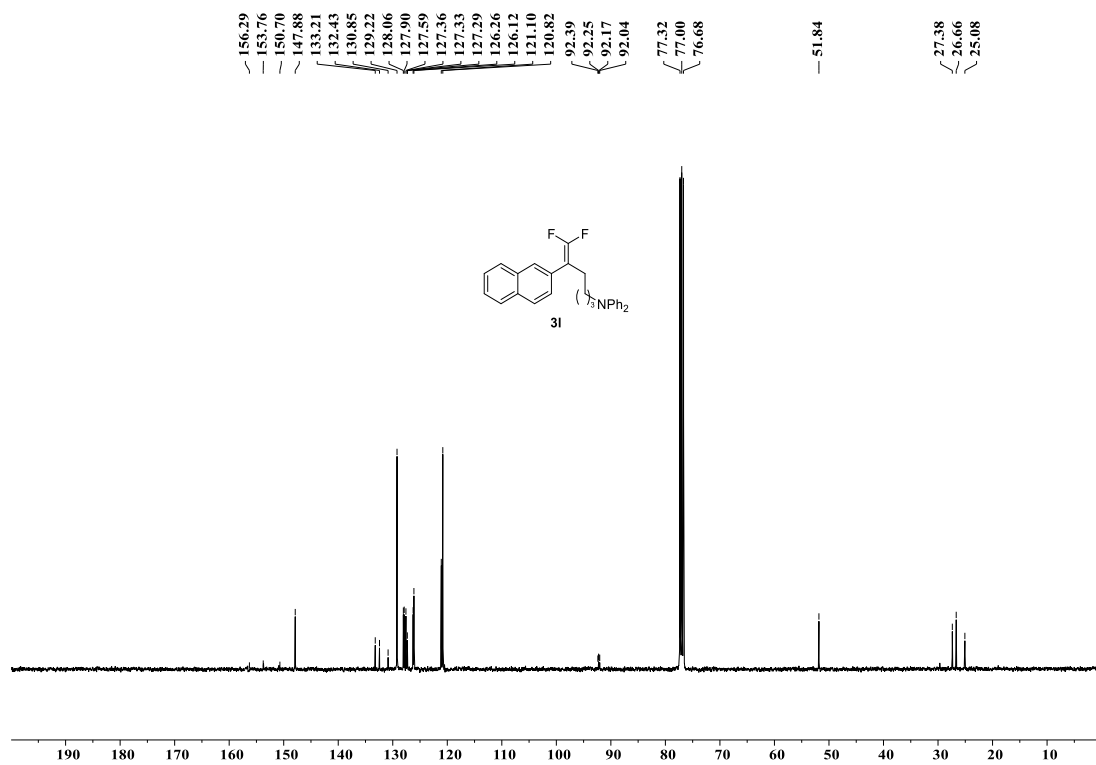
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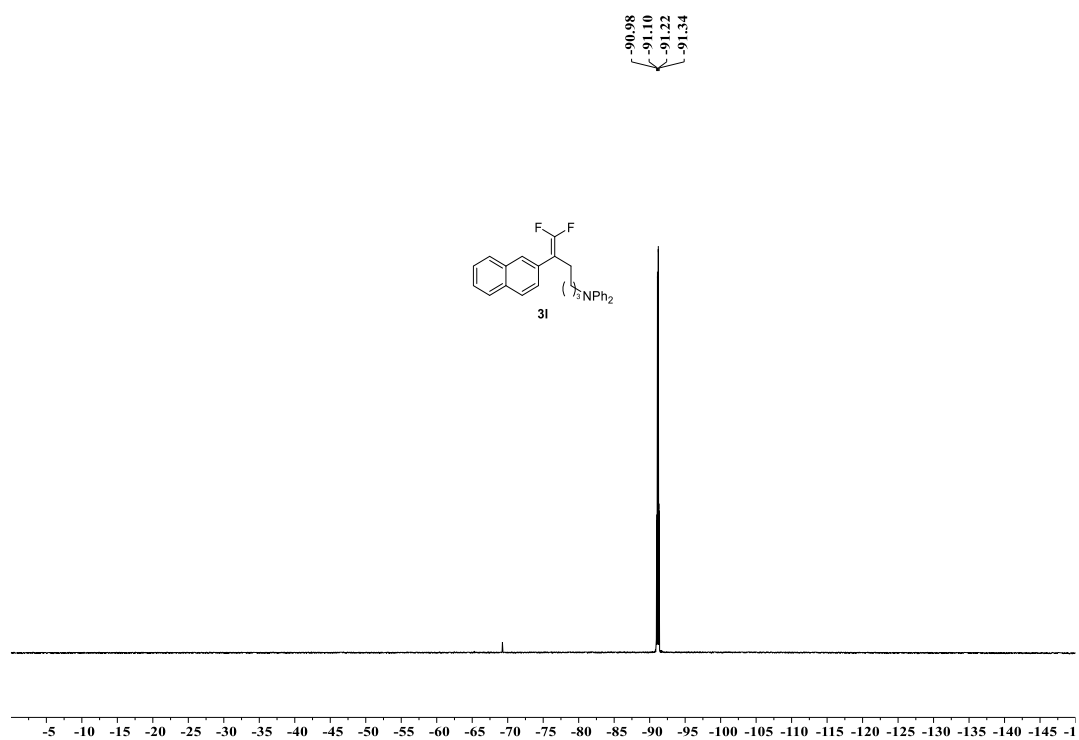
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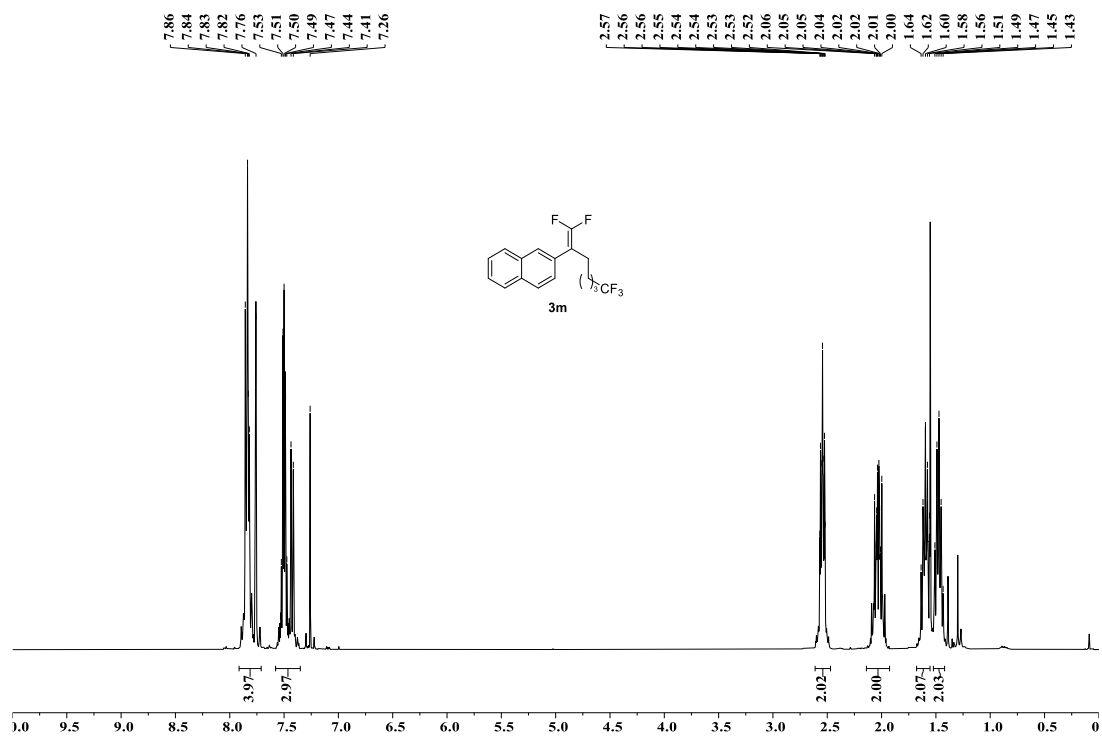
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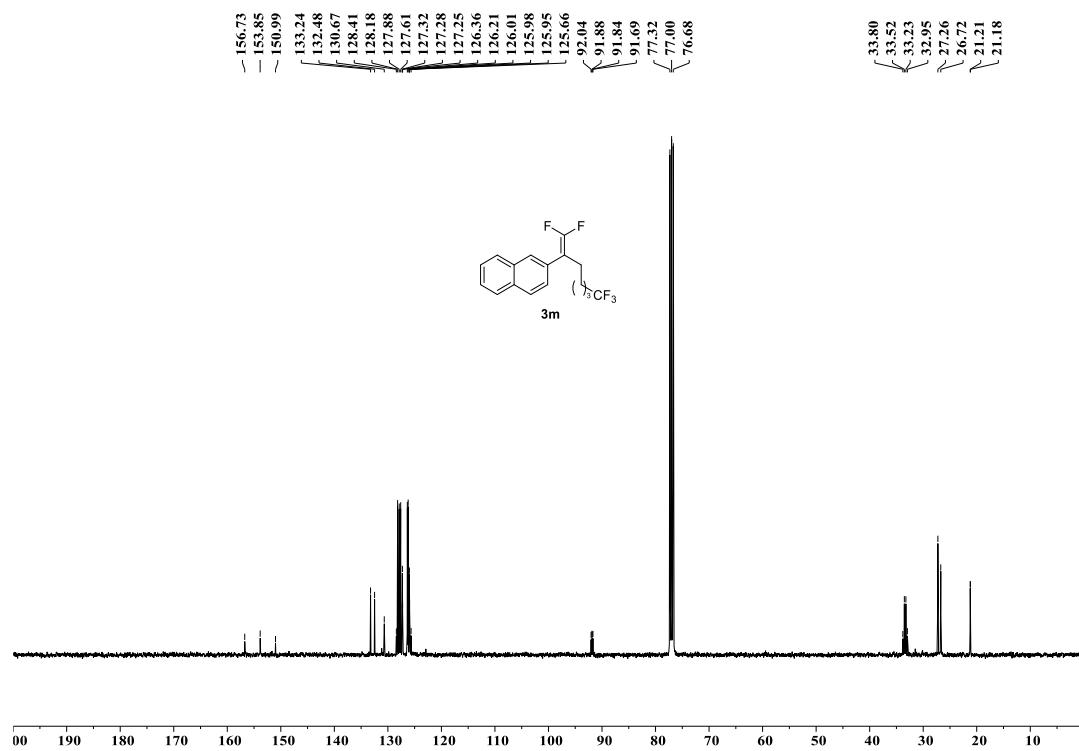
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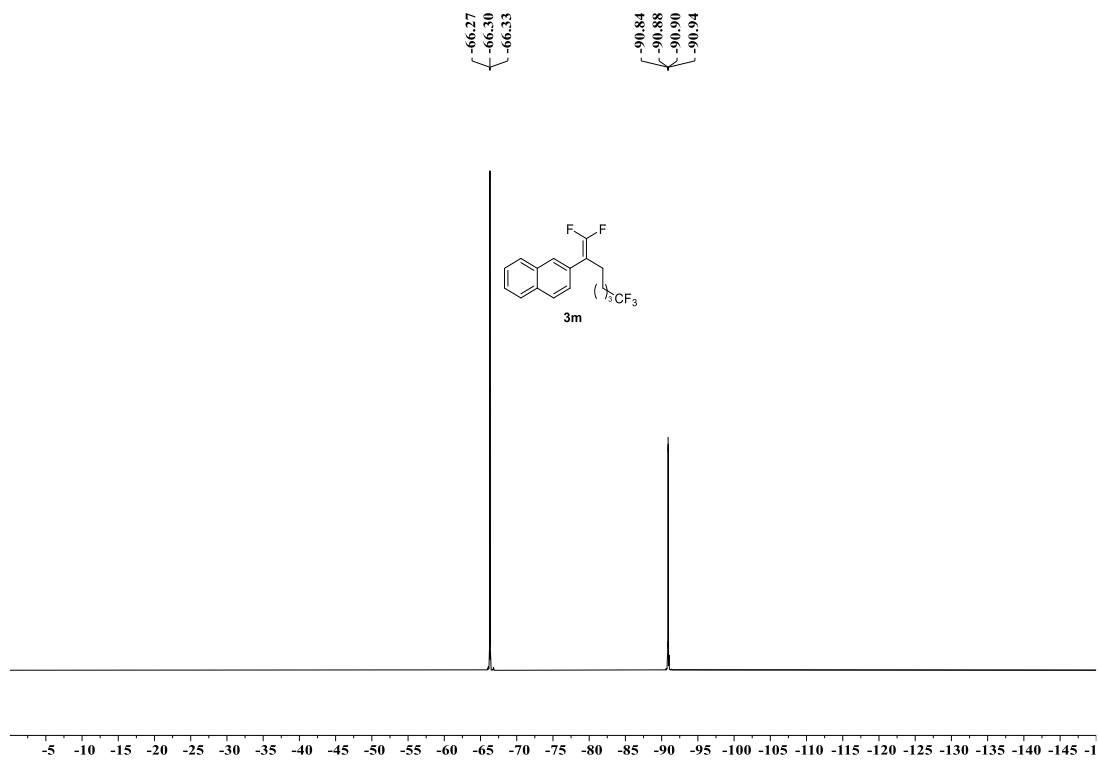
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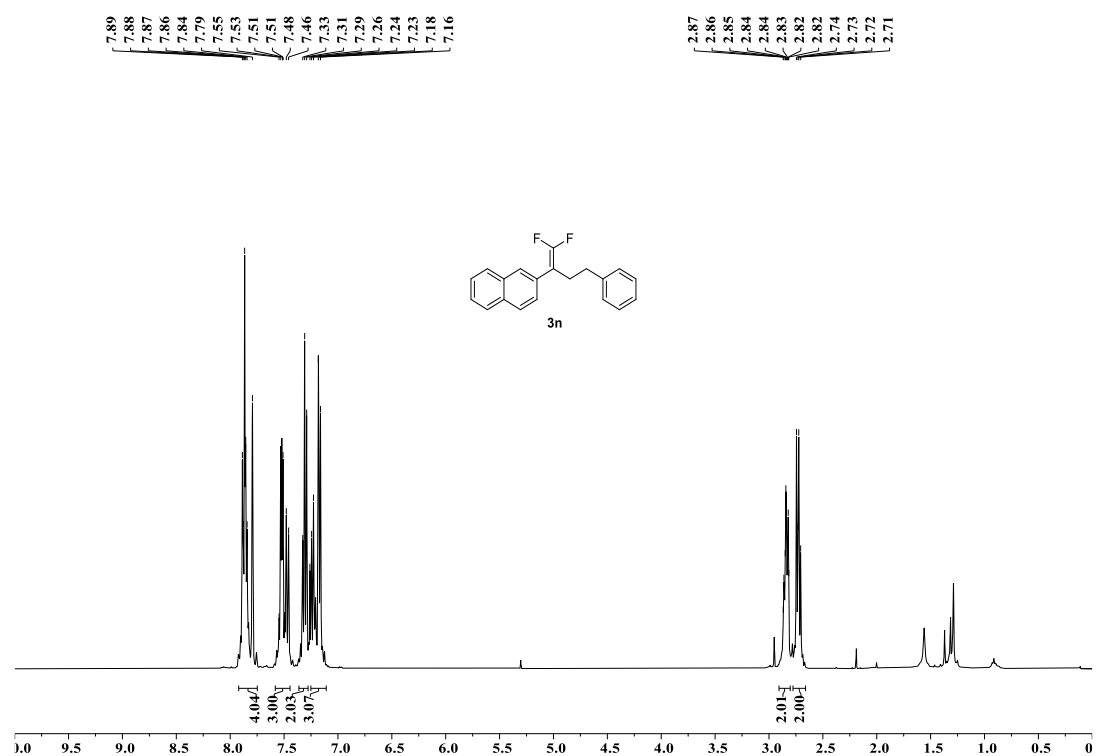
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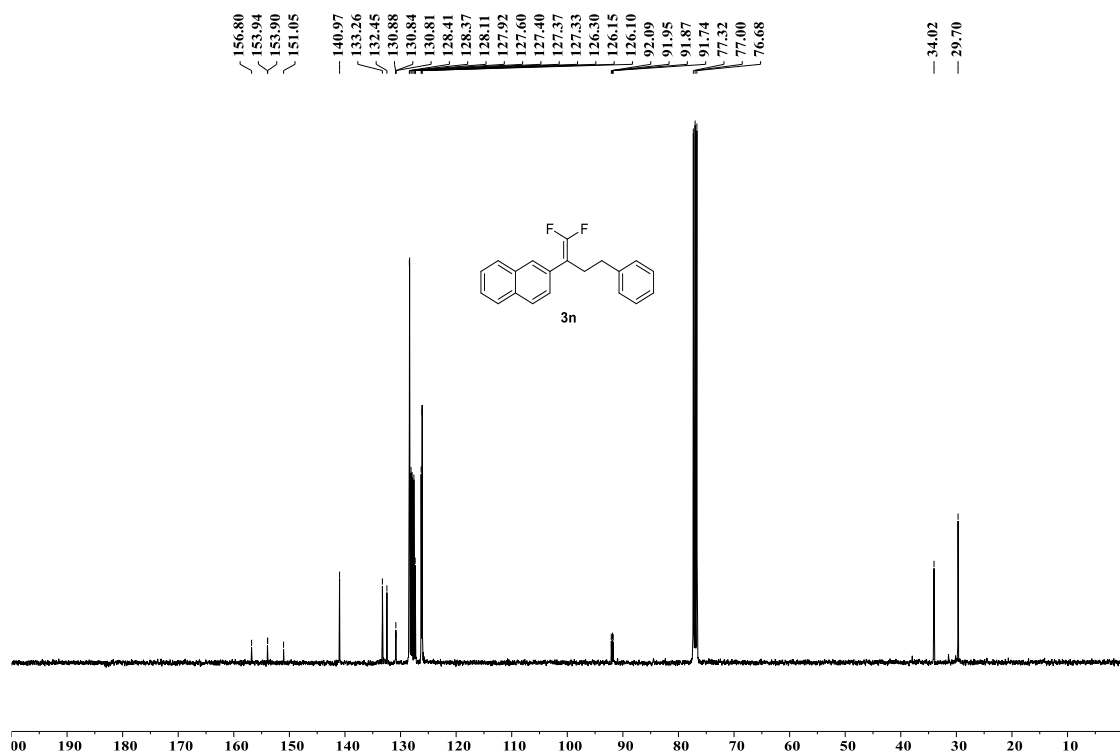
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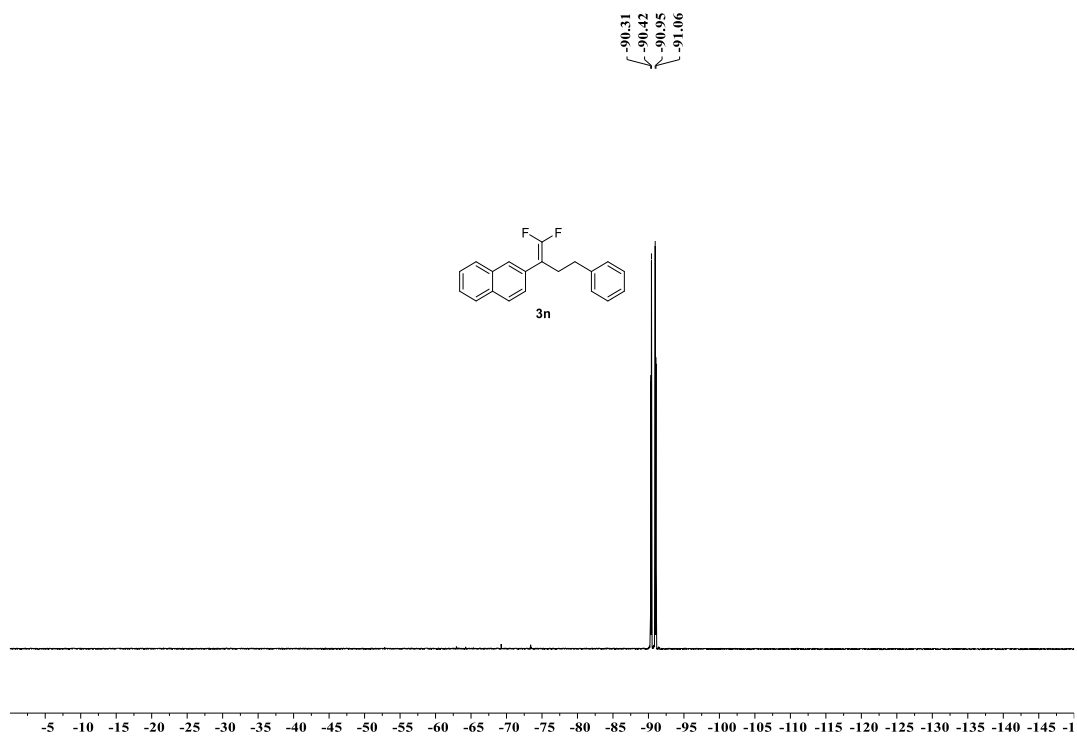
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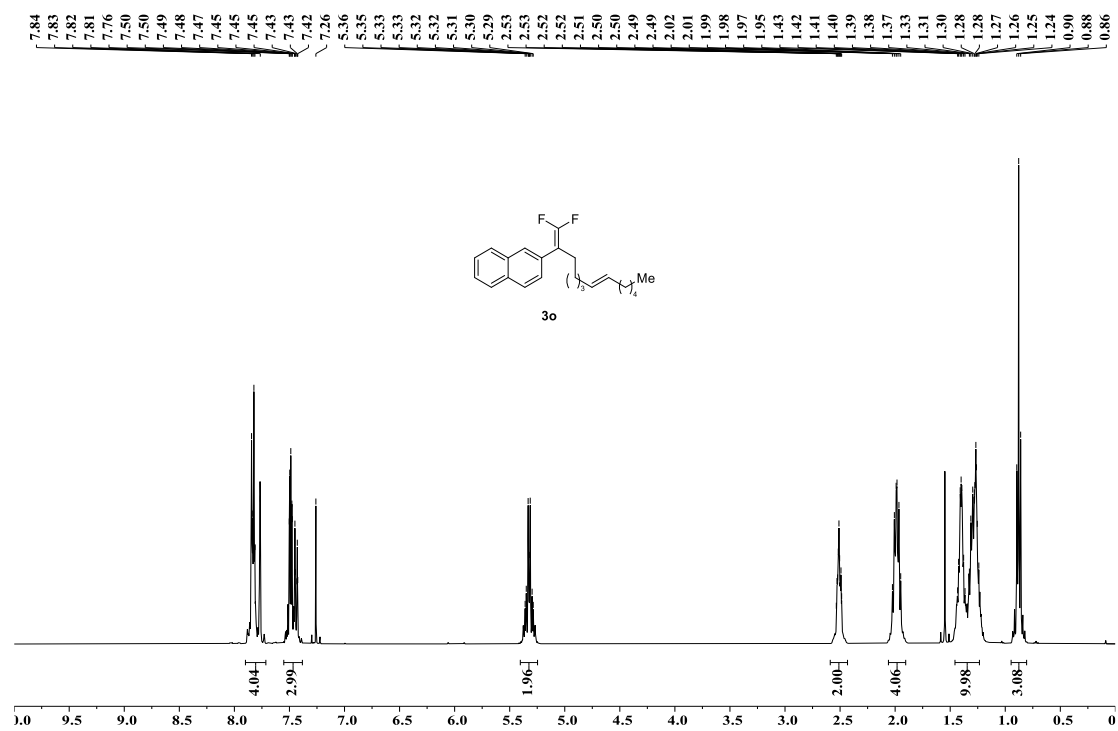
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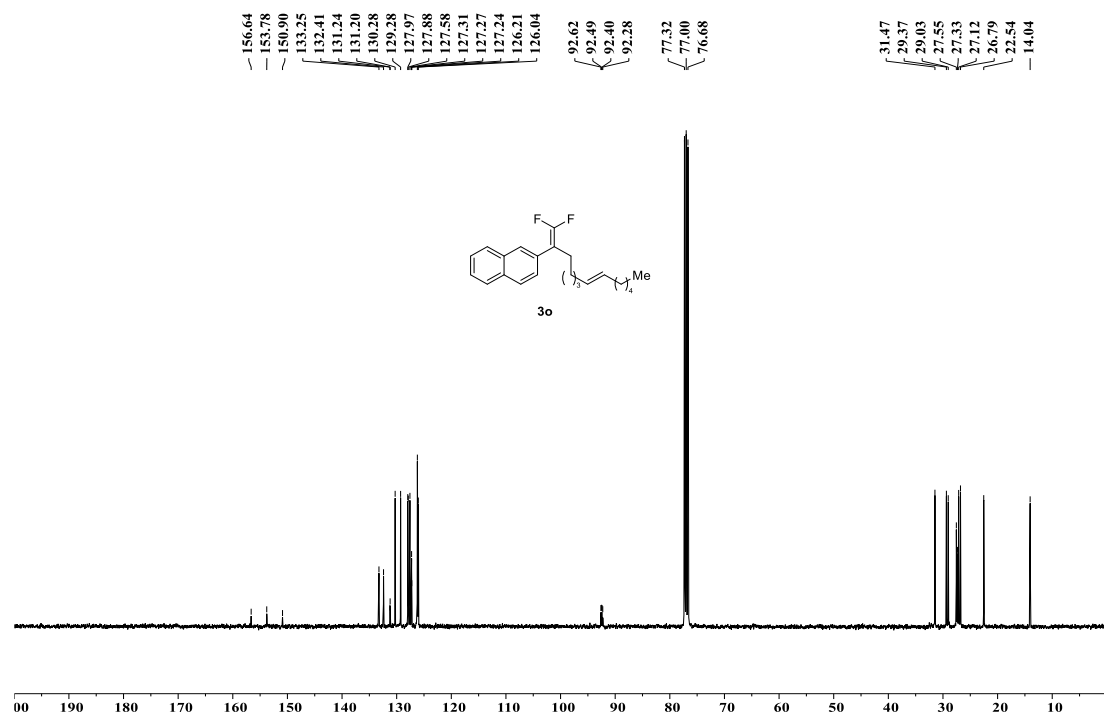
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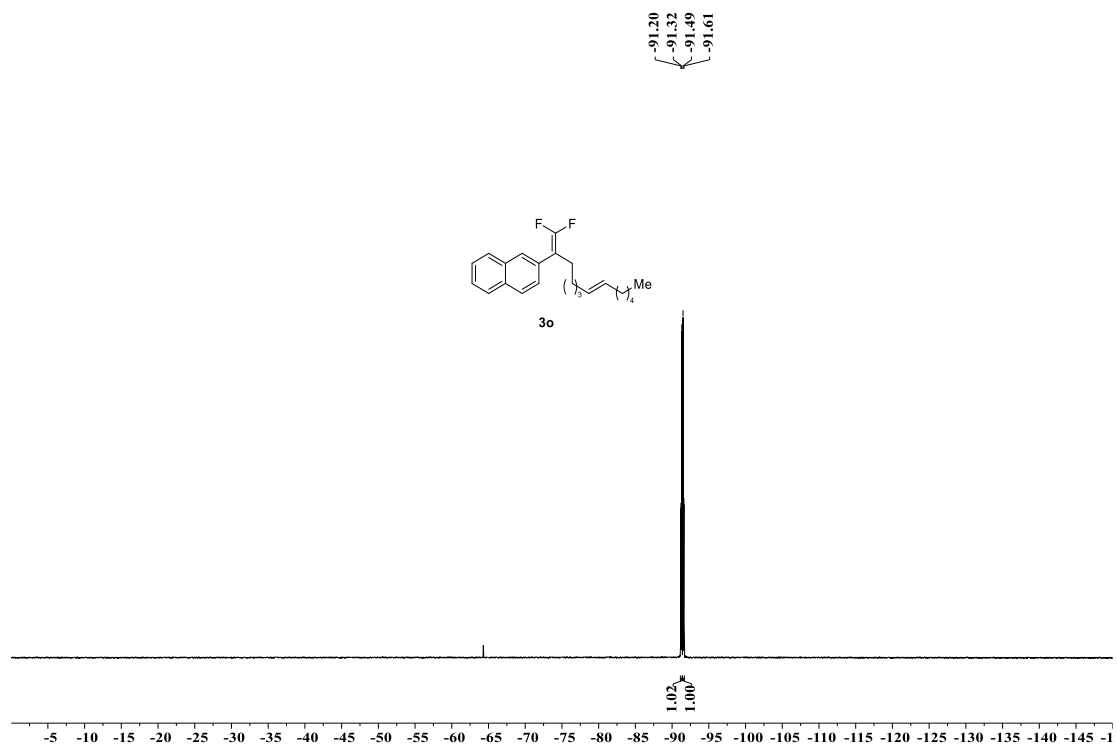
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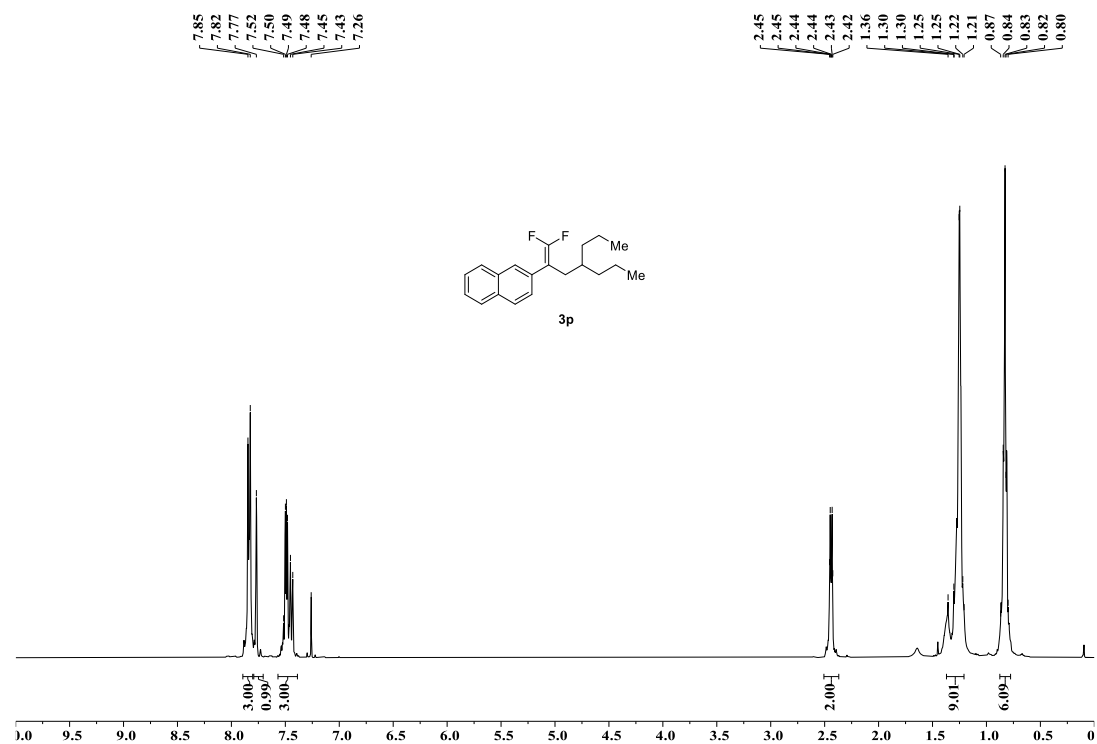
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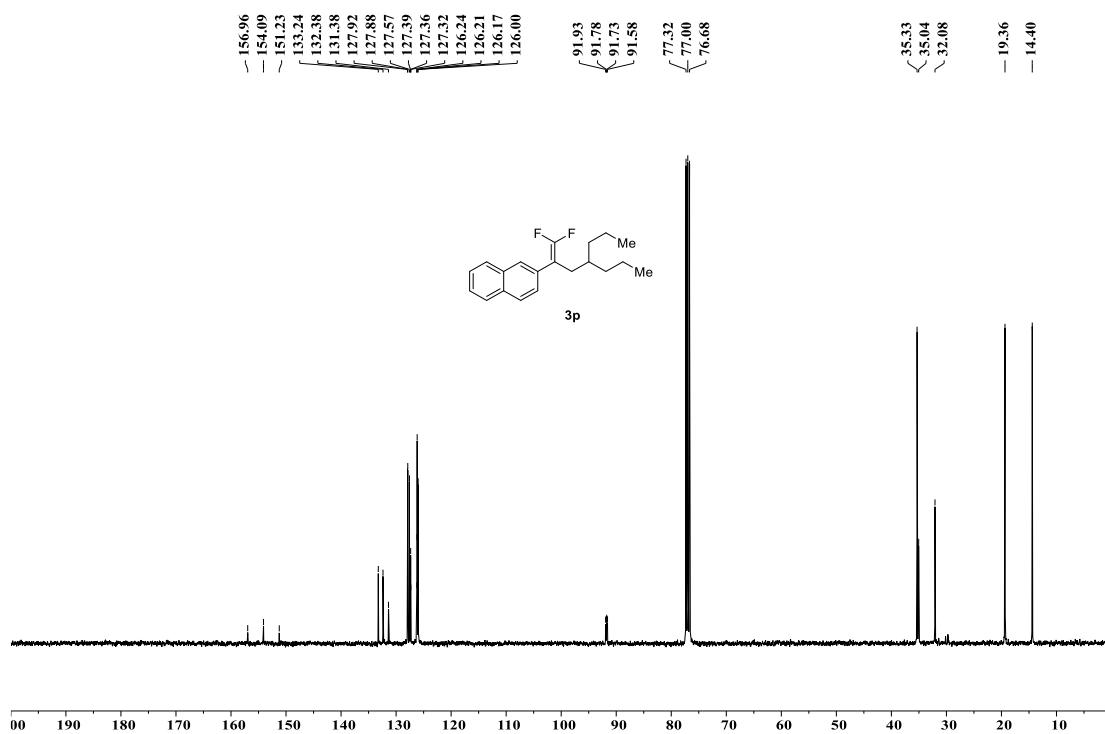
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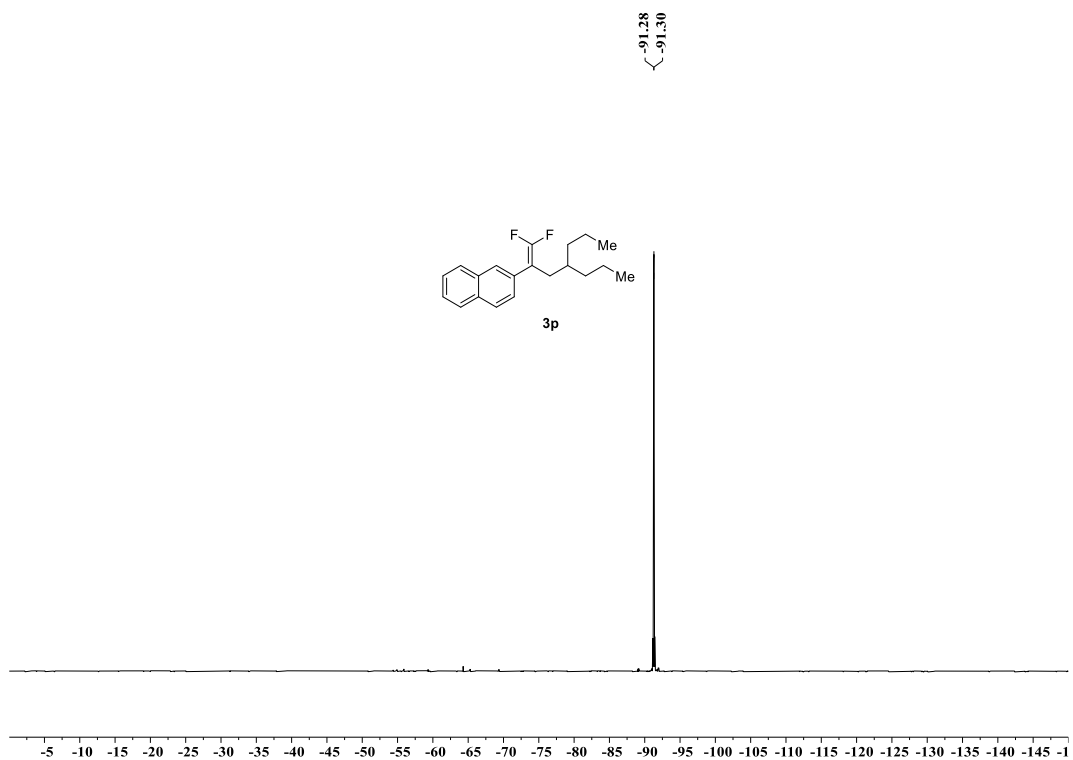
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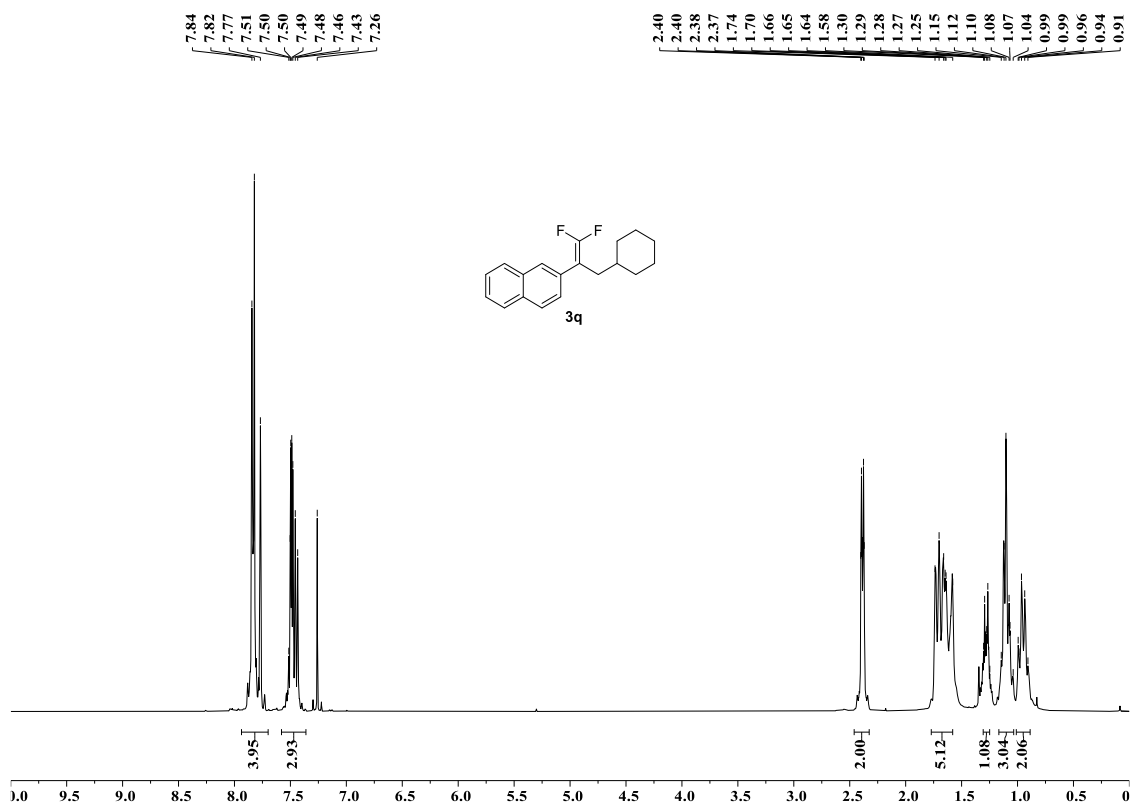
^{13}C NMR (100 MHz, CDCl_3)



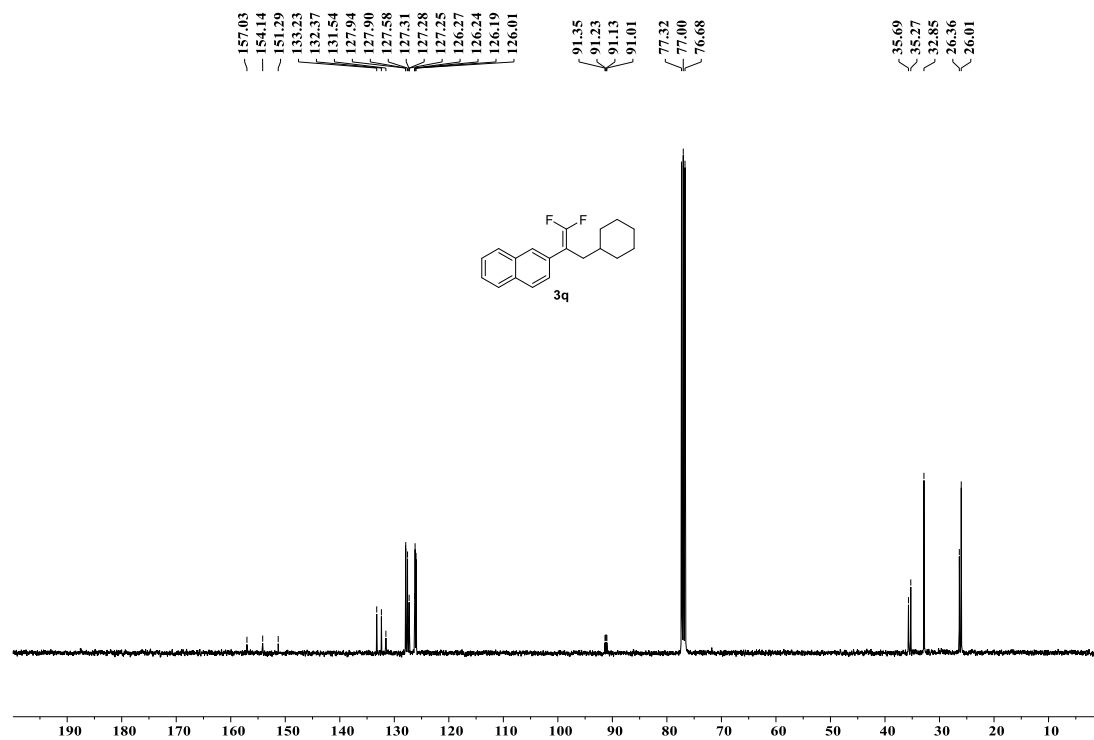
^{19}F NMR (376 MHz, CDCl_3)



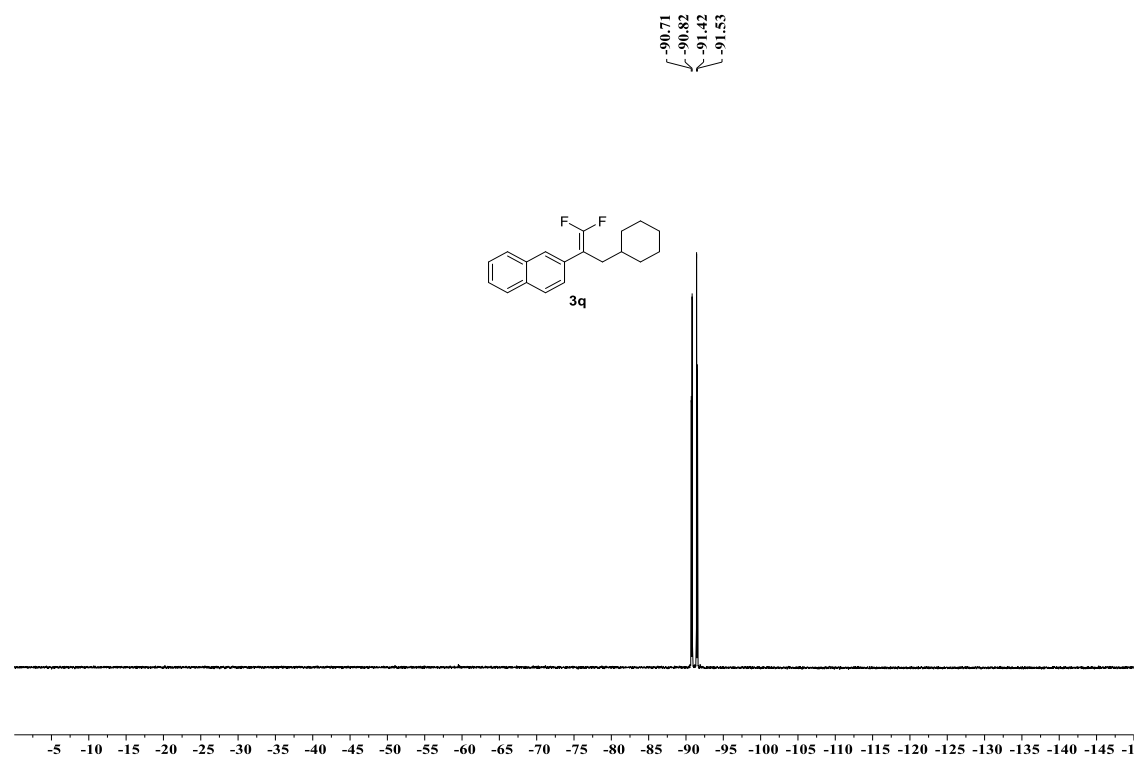
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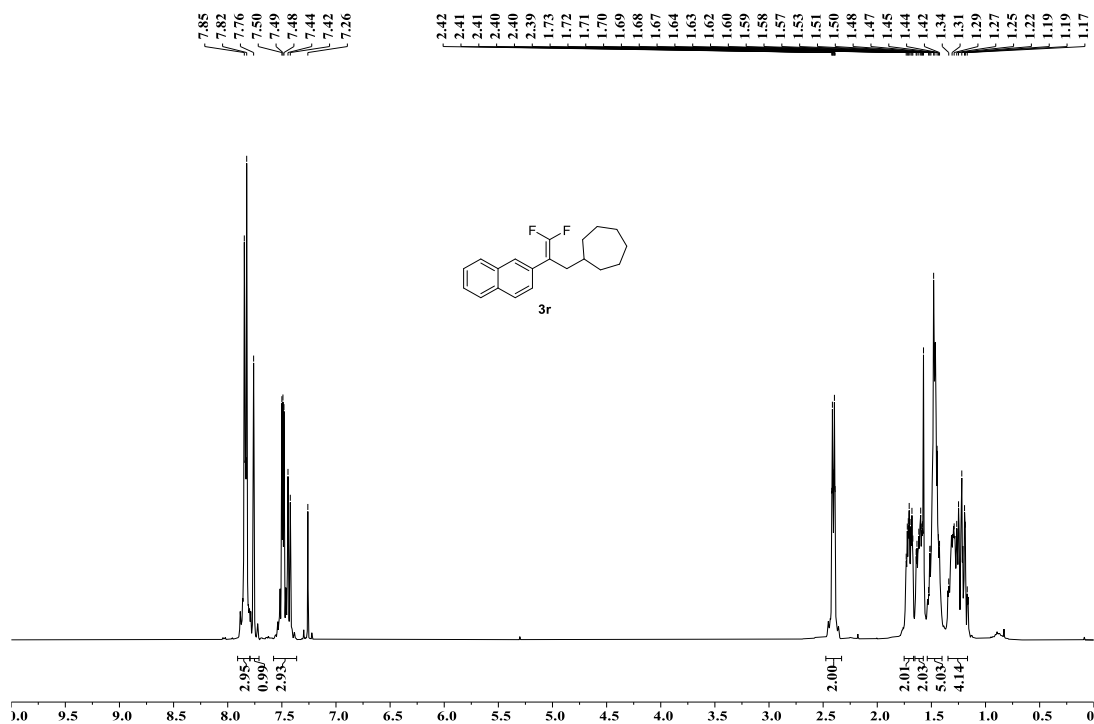
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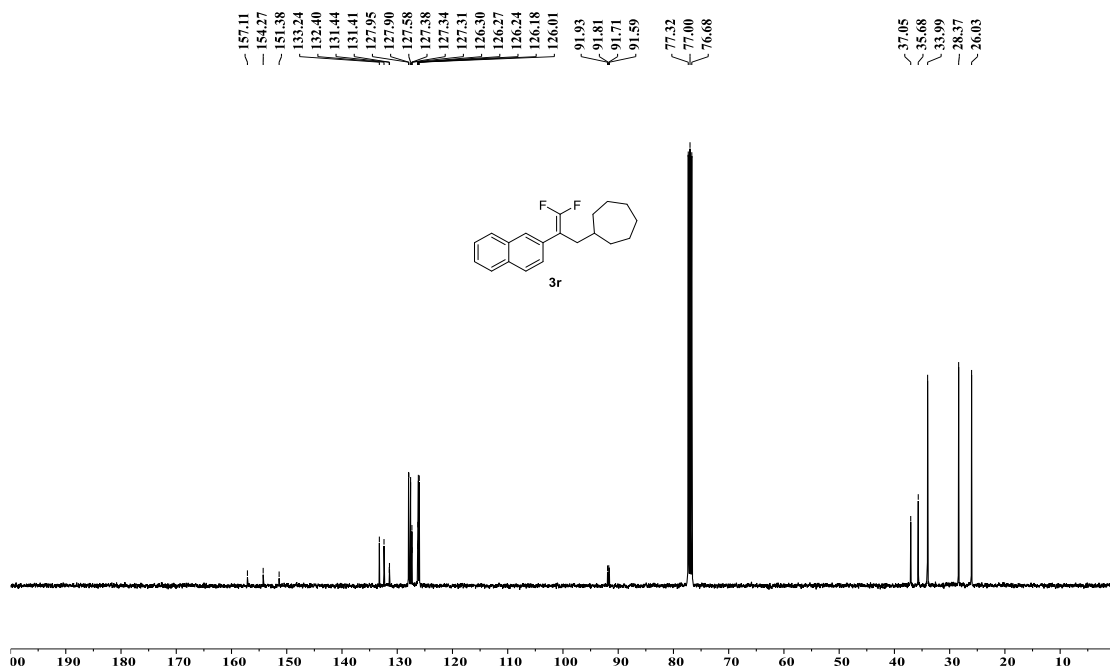
^{19}F NMR (376 MHz, CDCl_3)



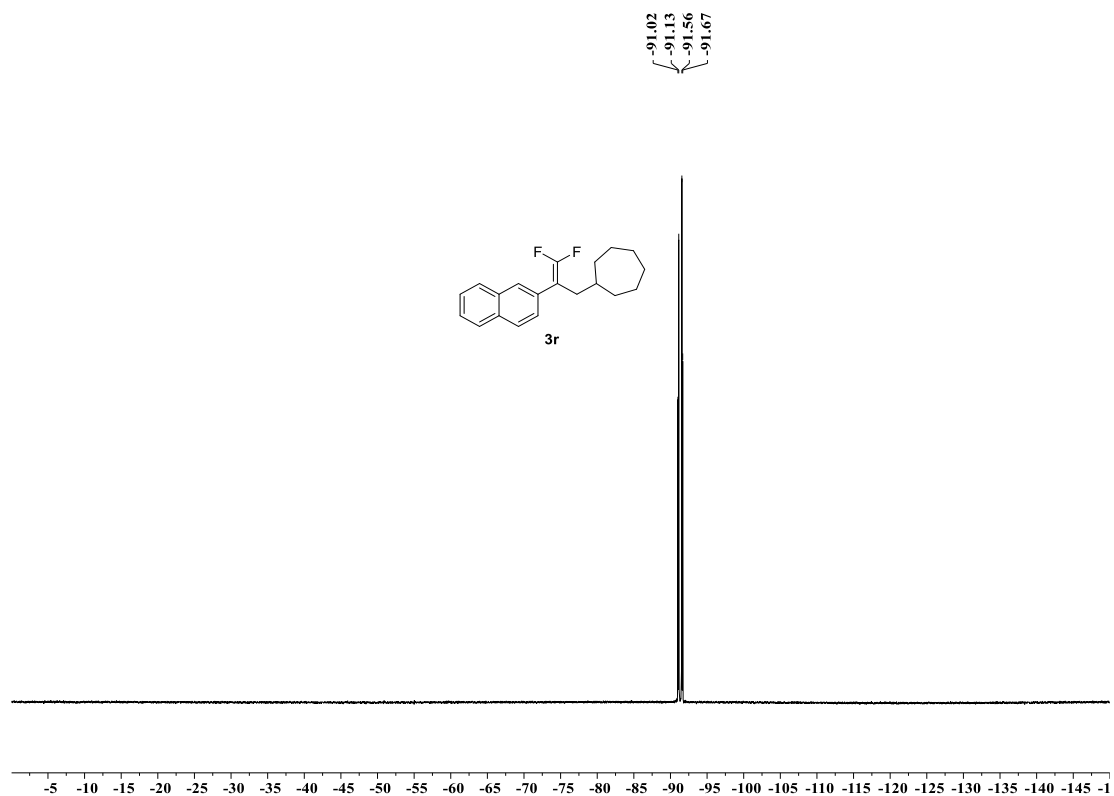
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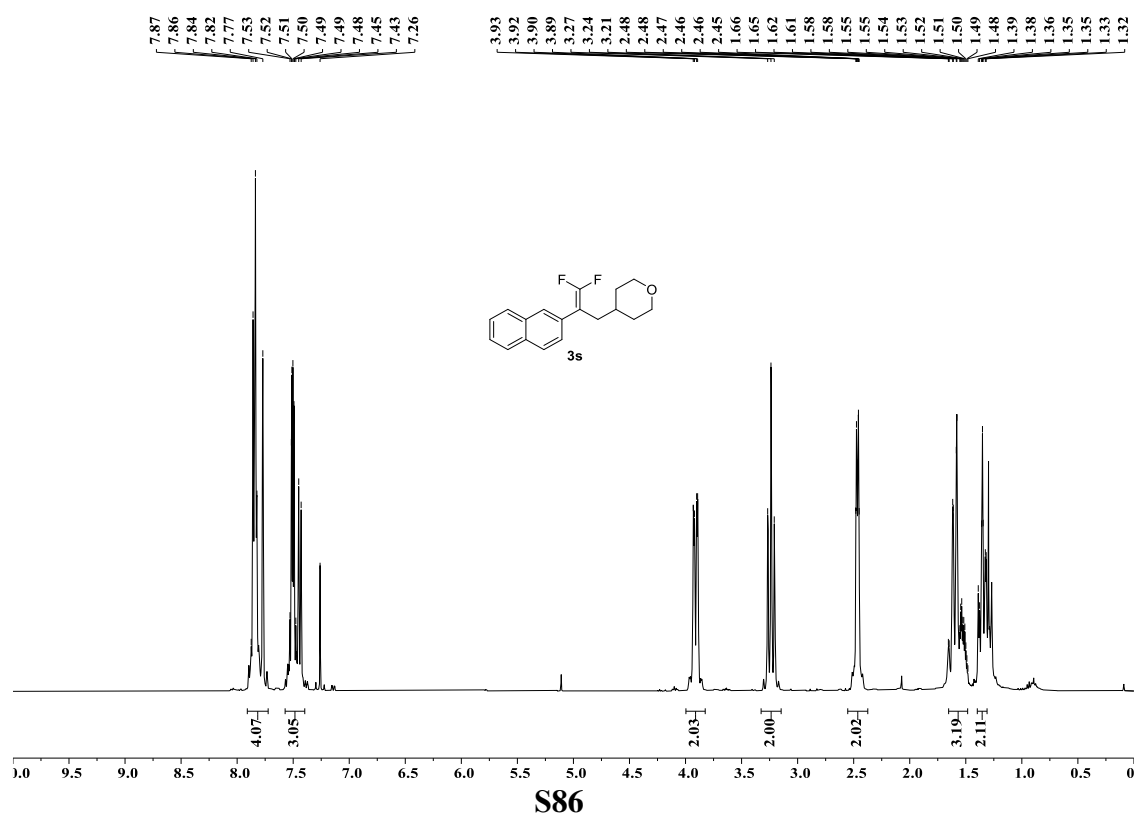
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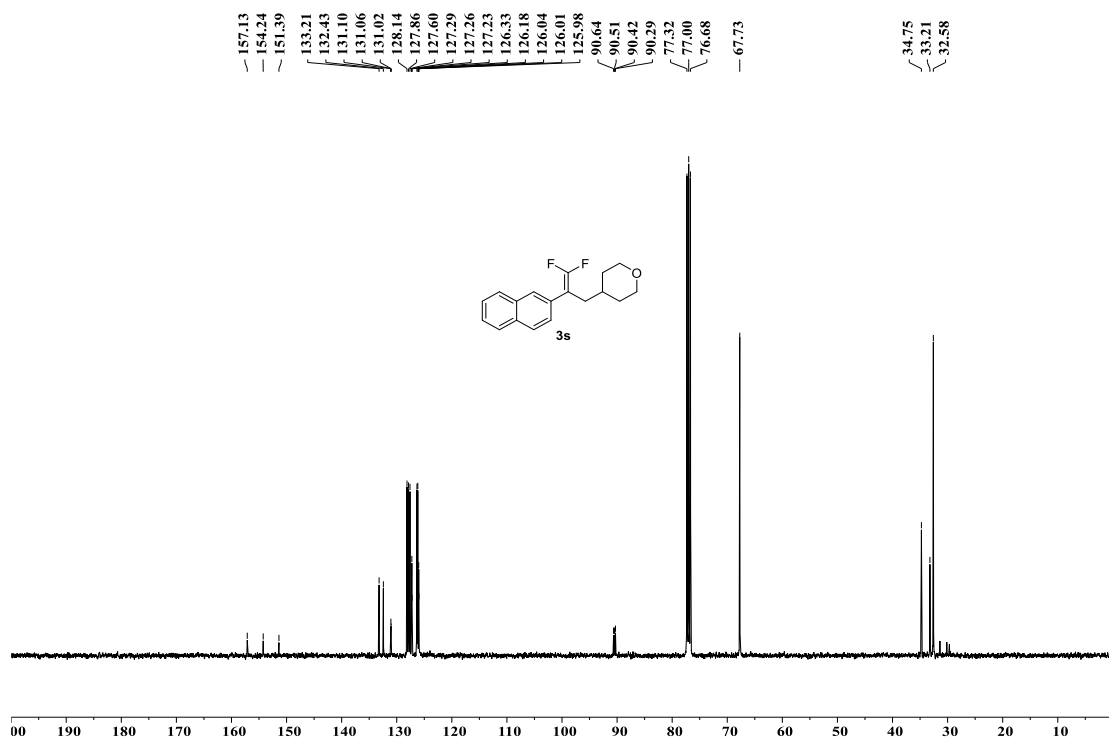
^{19}F NMR (376 MHz, CDCl_3)



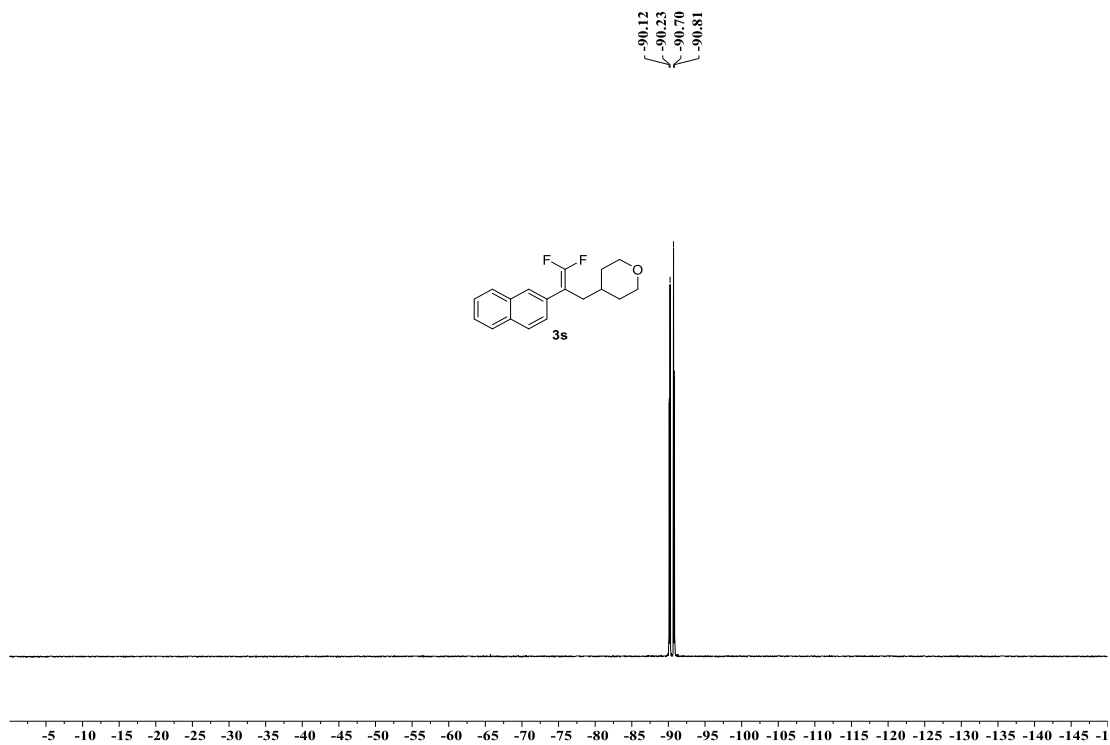
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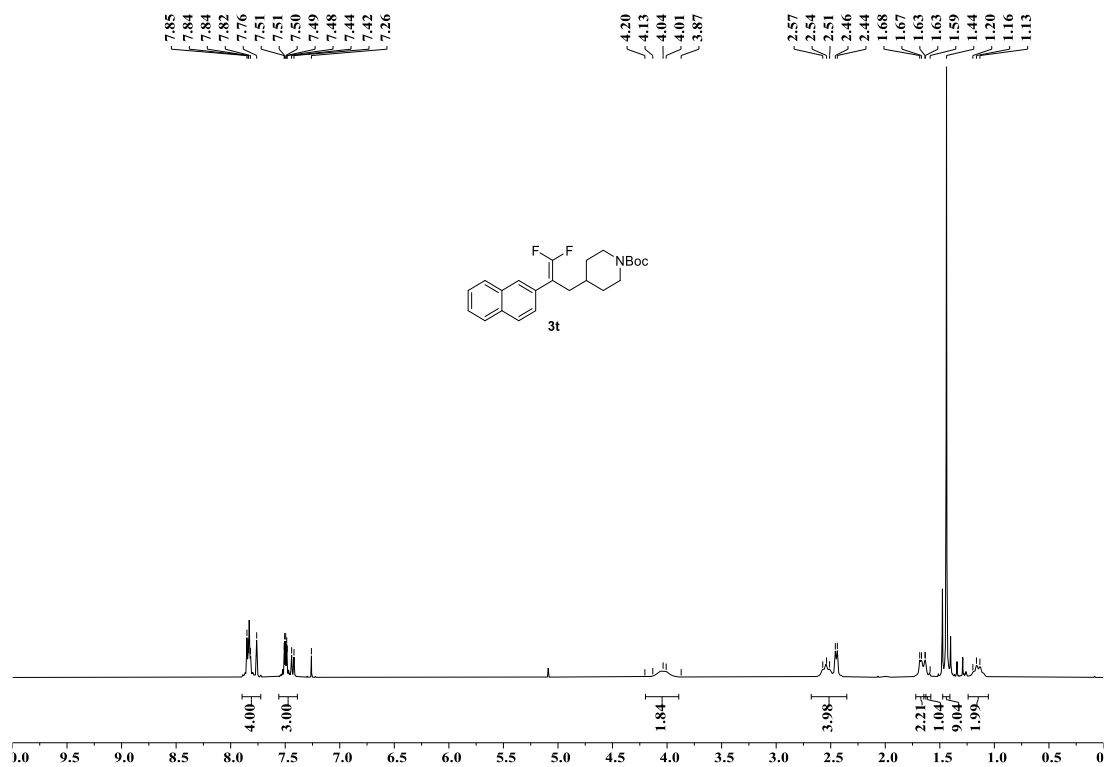
^{13}C NMR (100 MHz, CDCl_3)



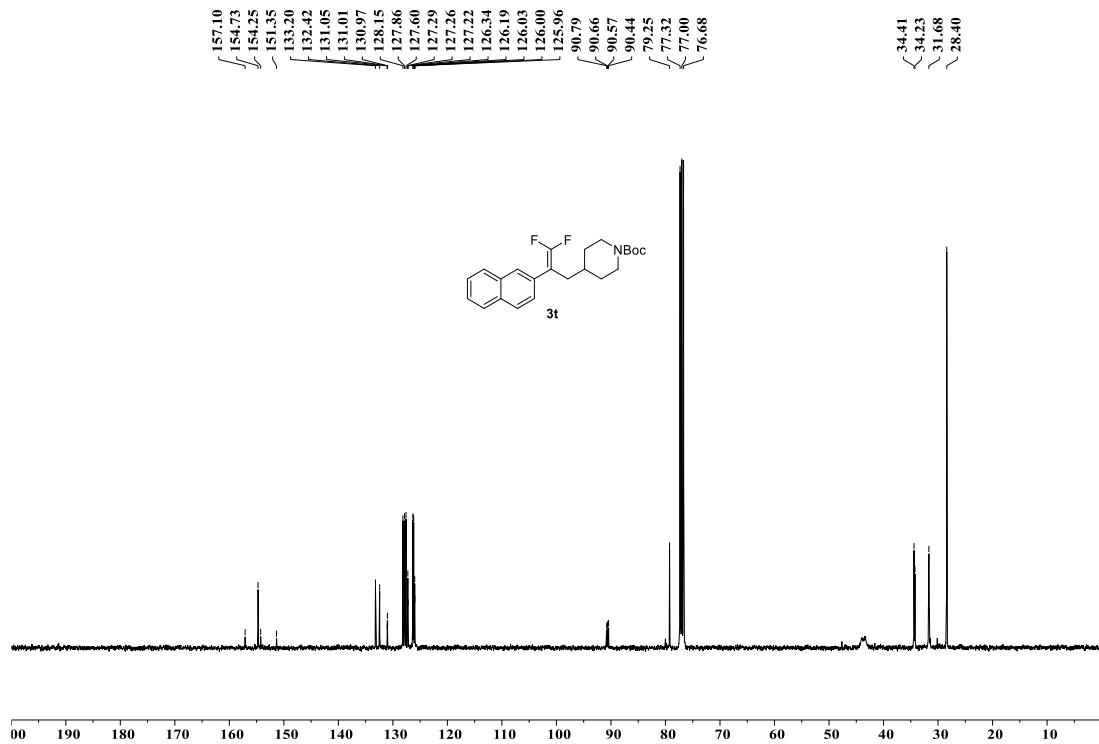
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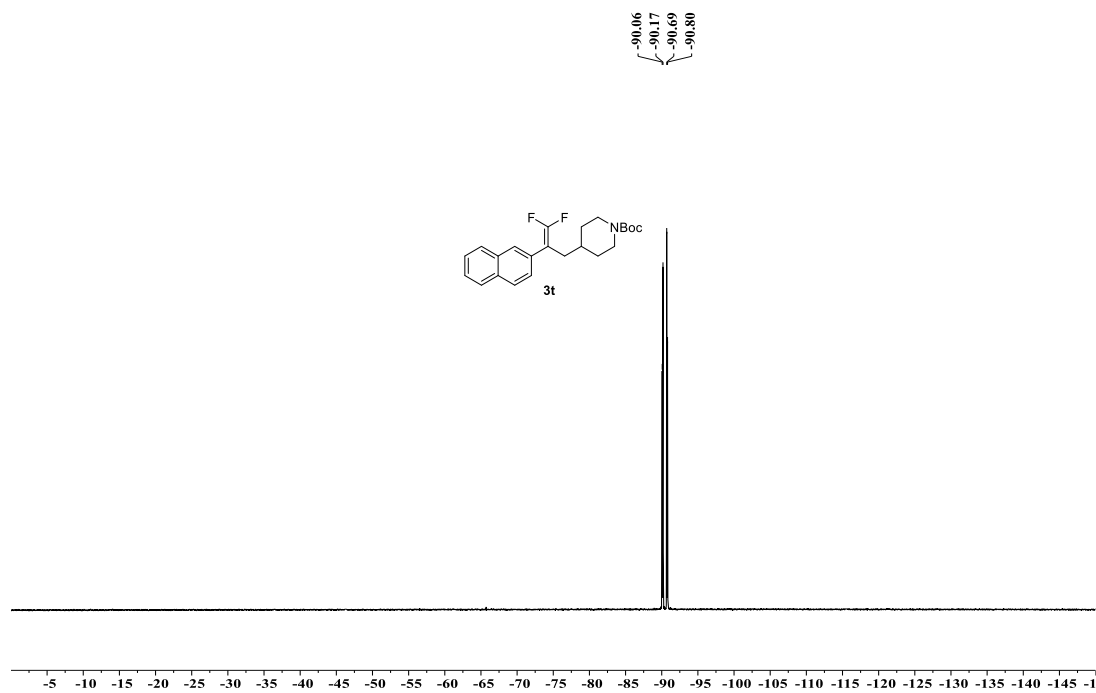
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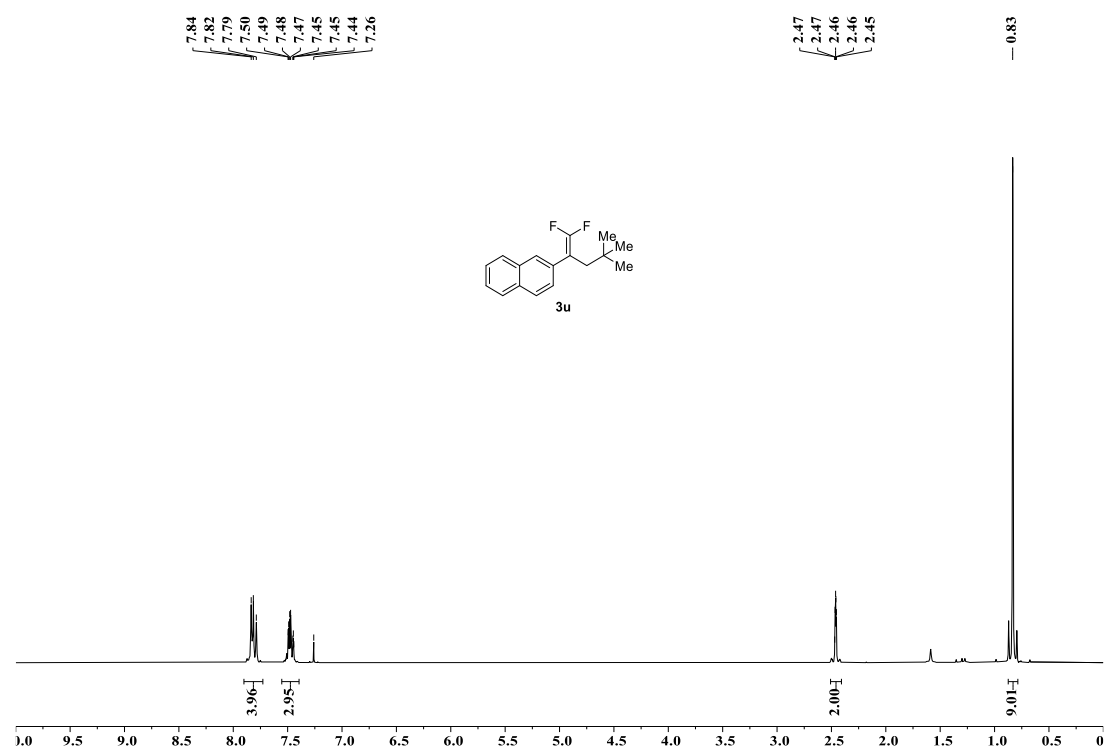
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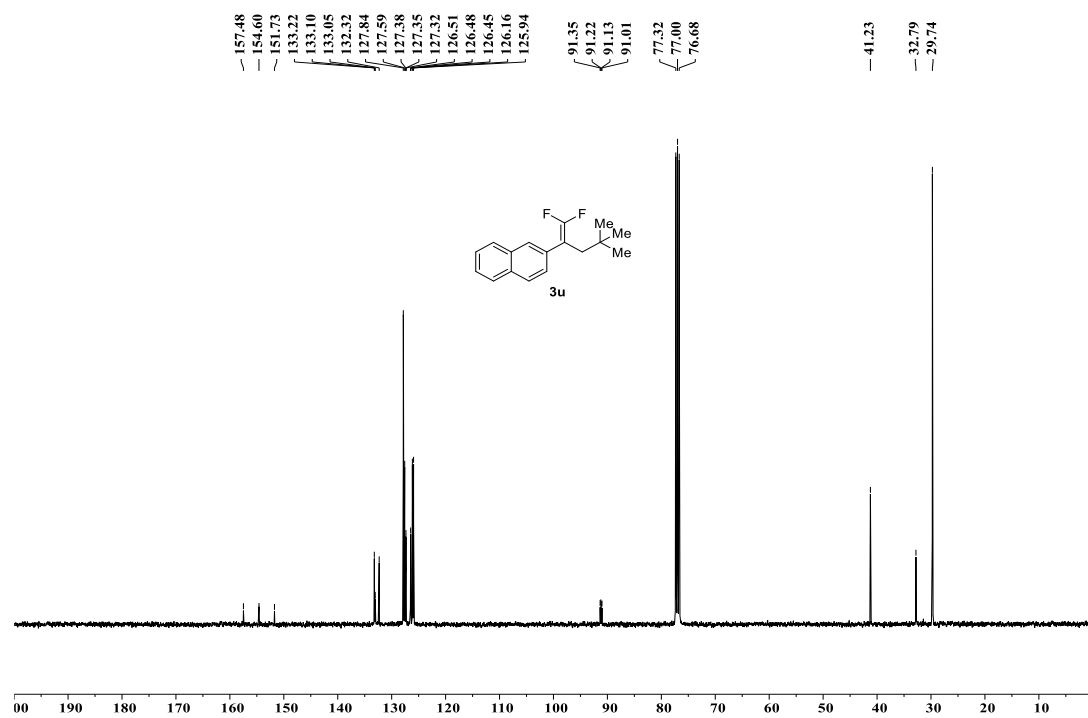
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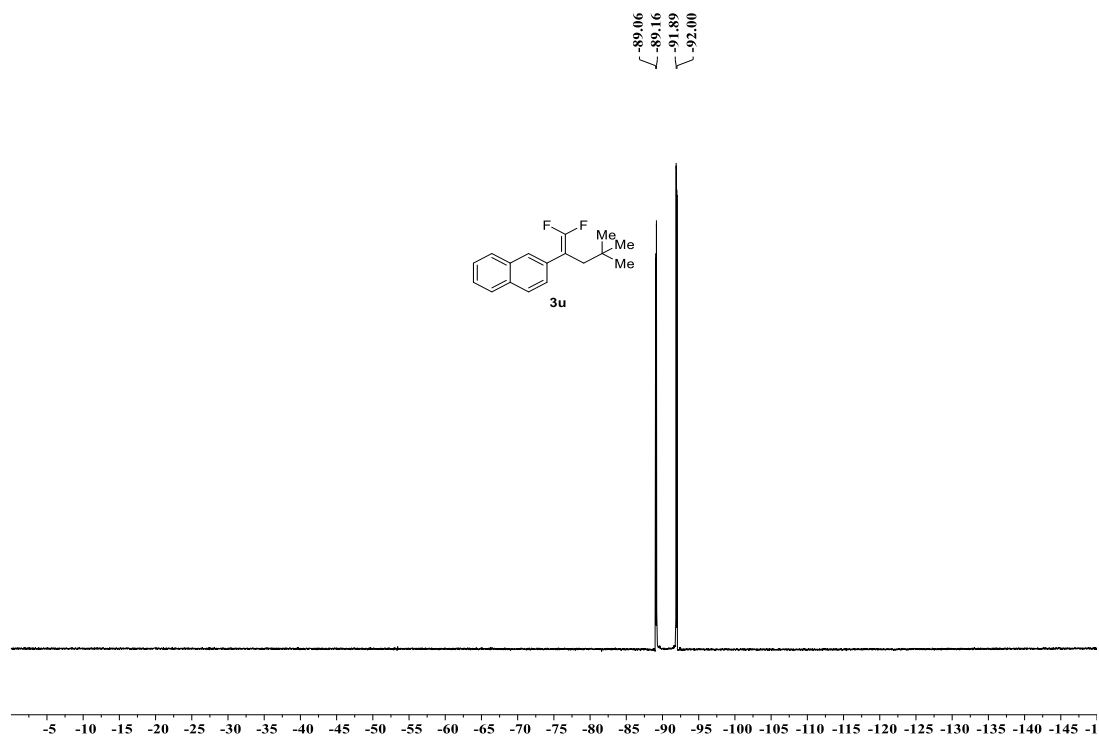
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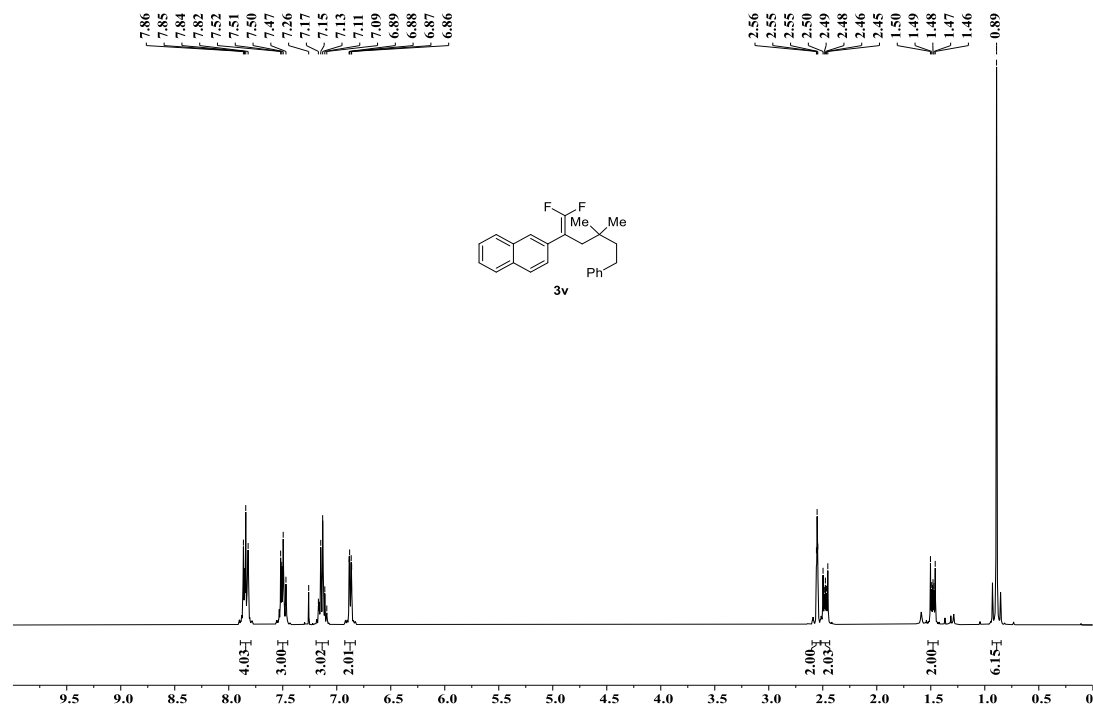
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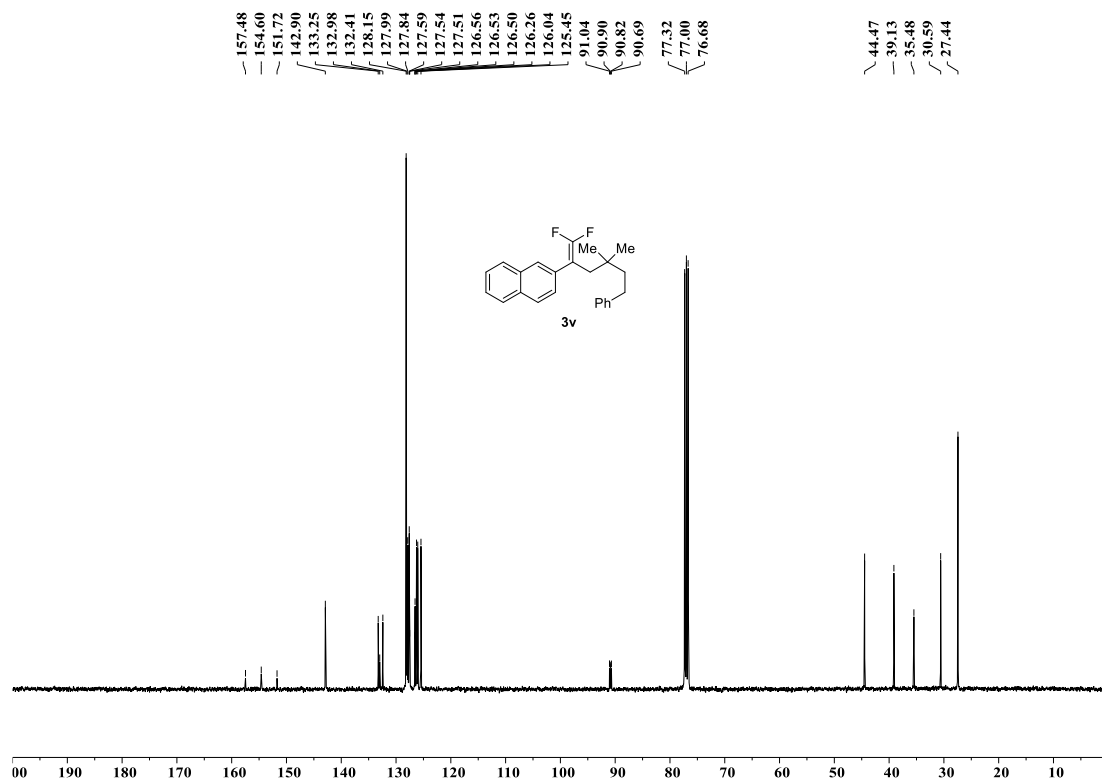
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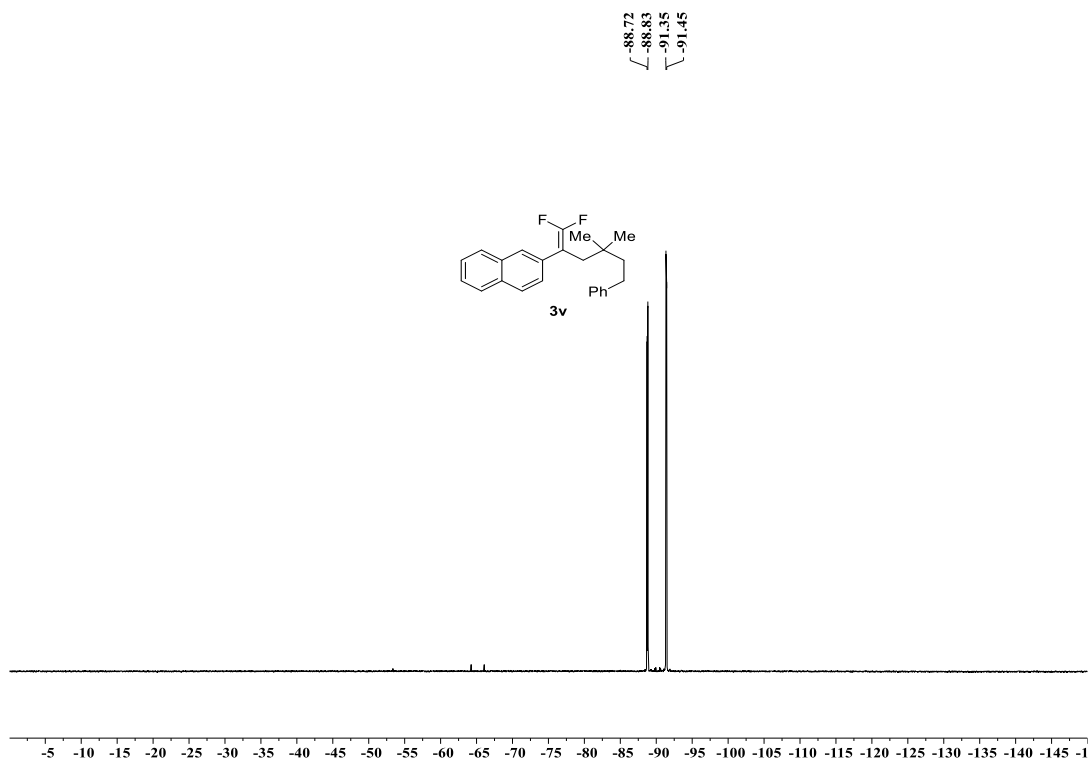
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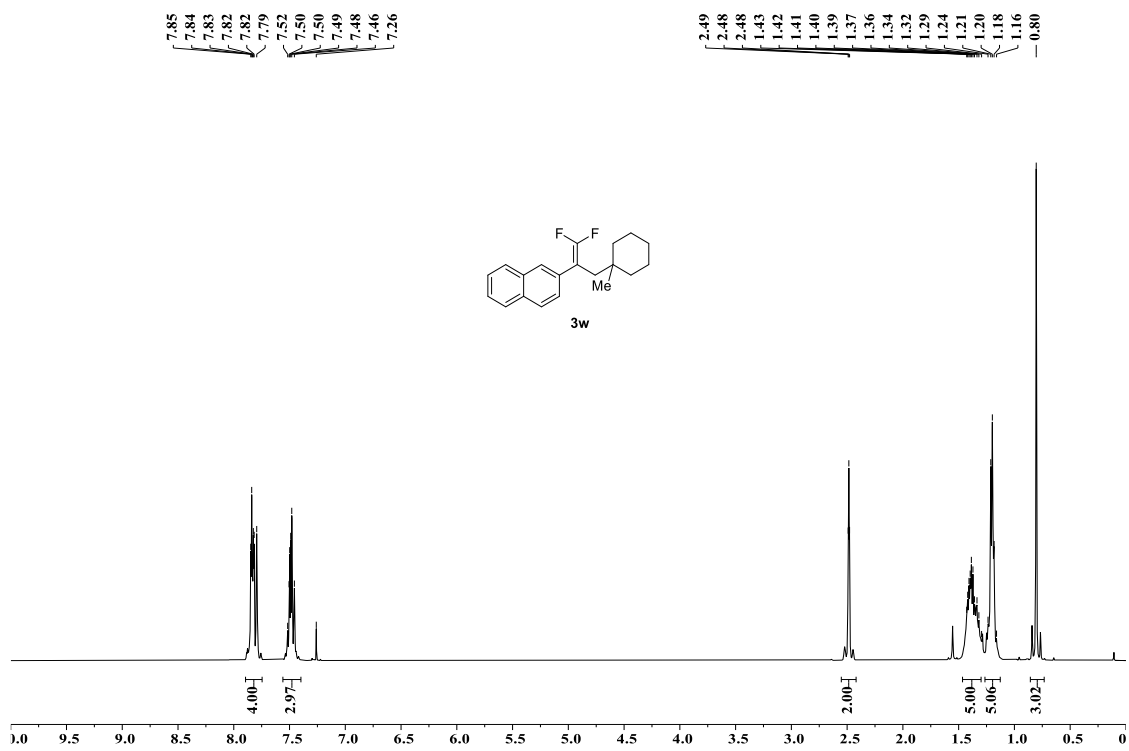
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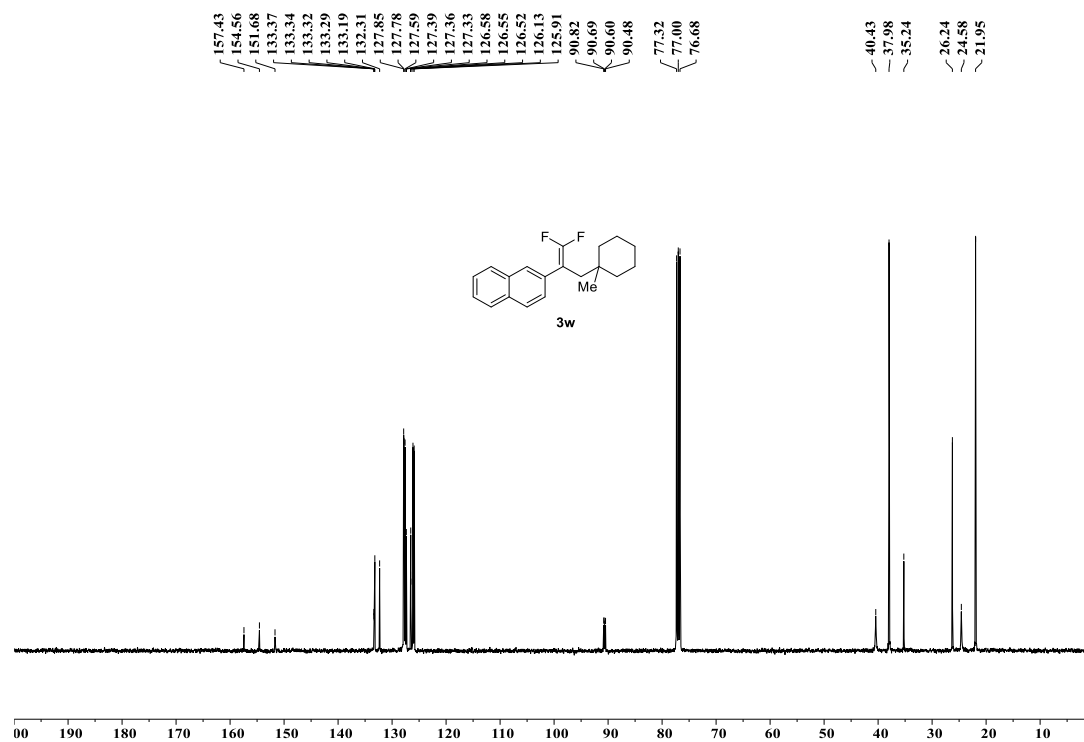
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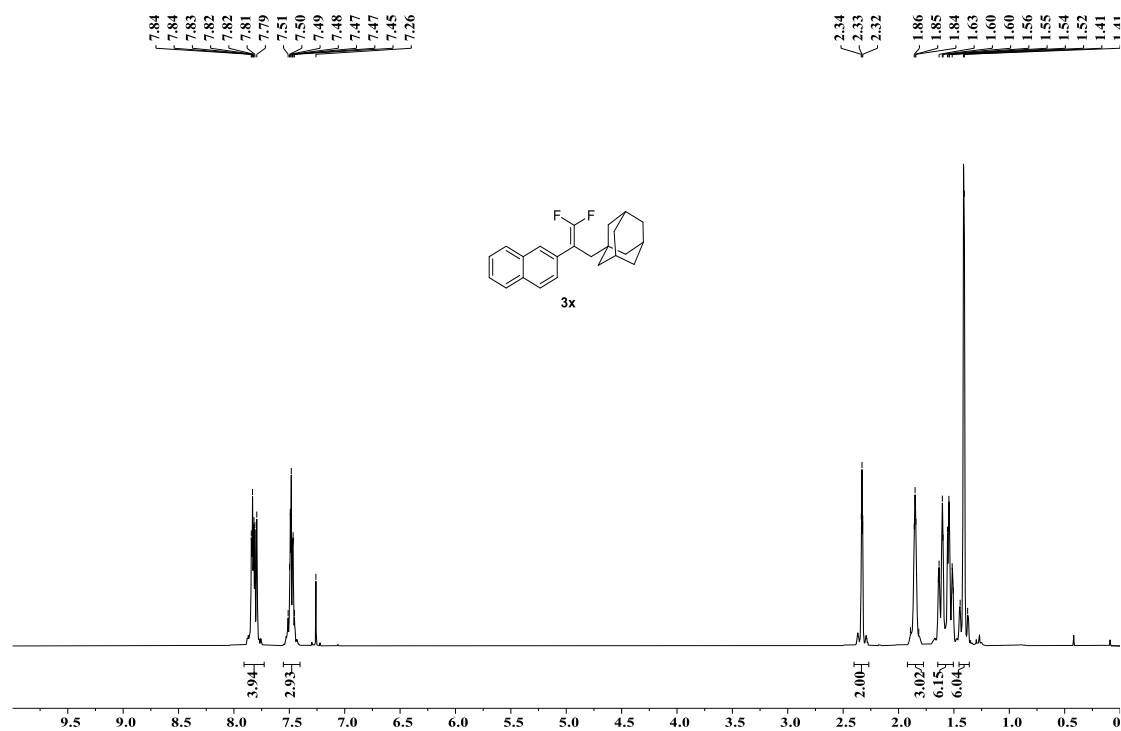
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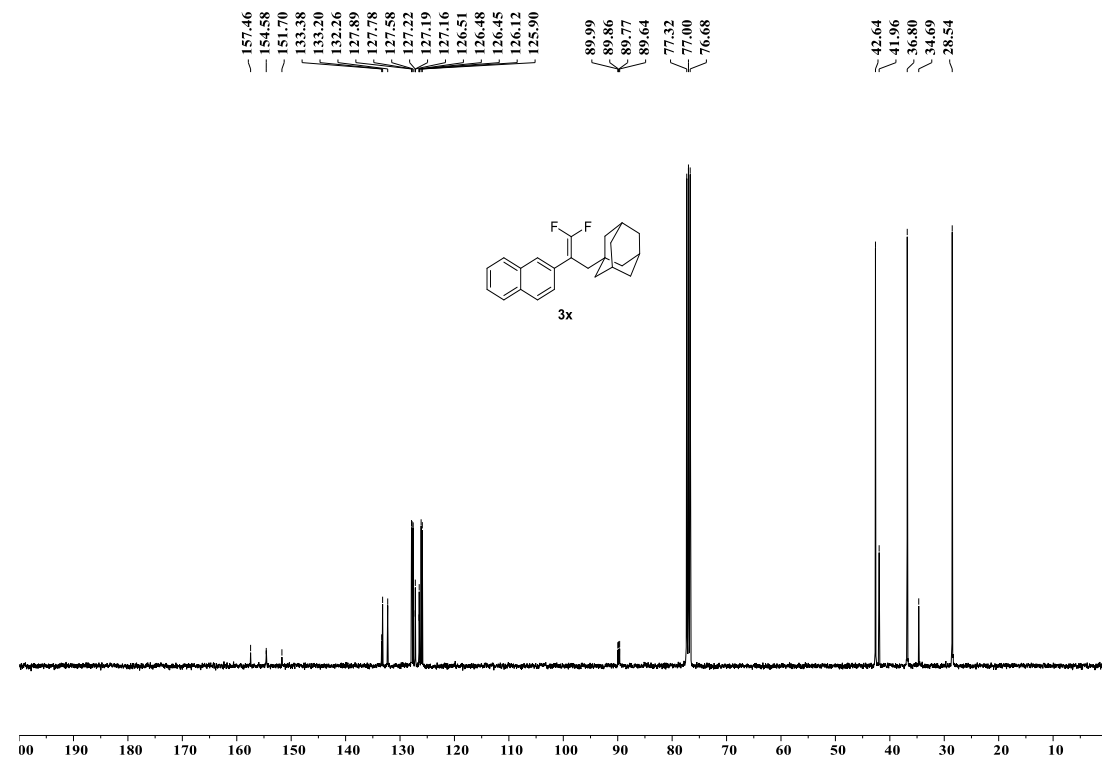
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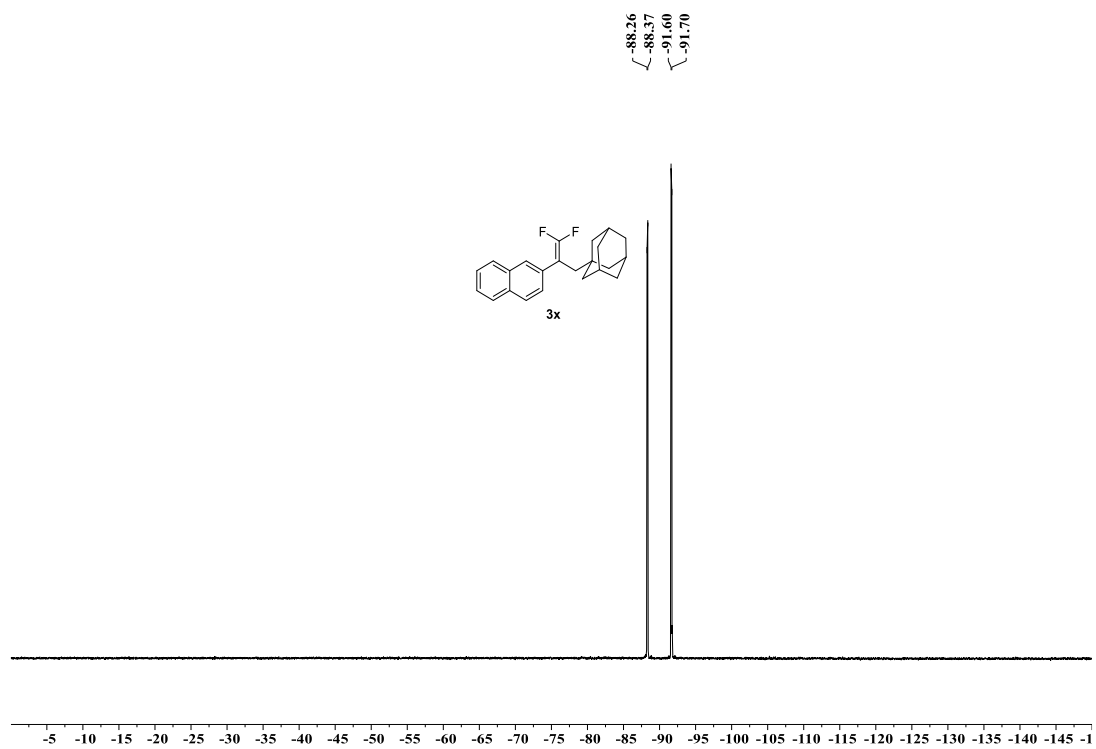
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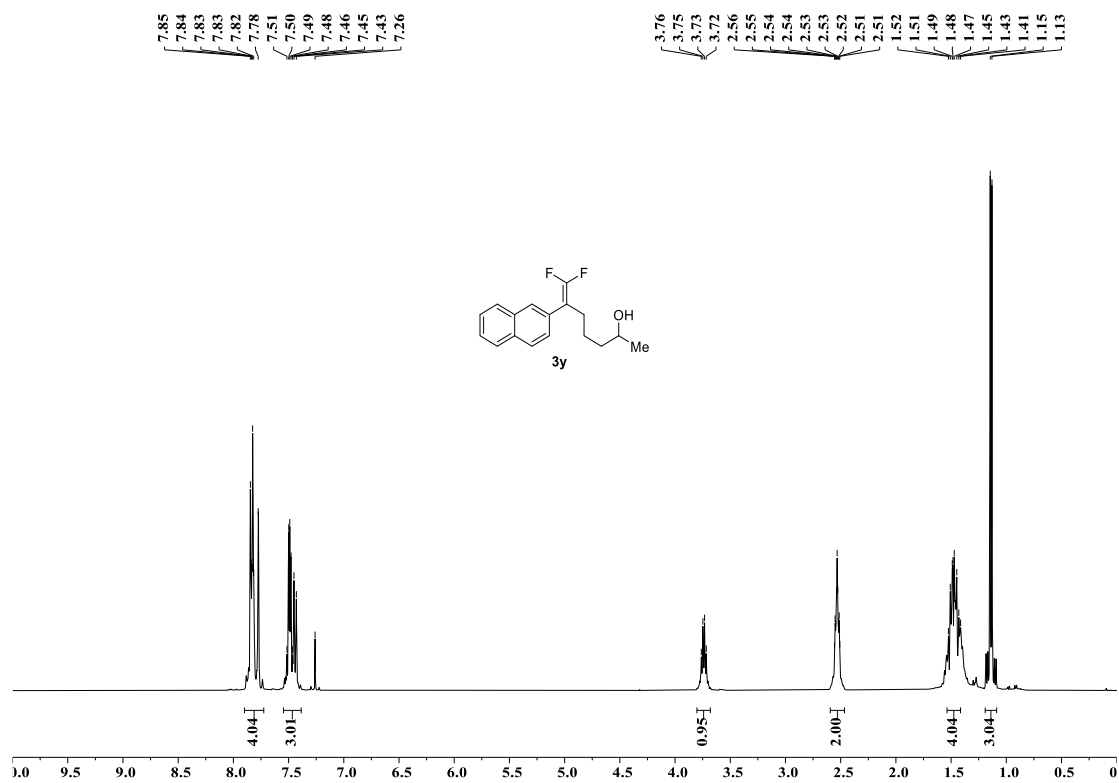
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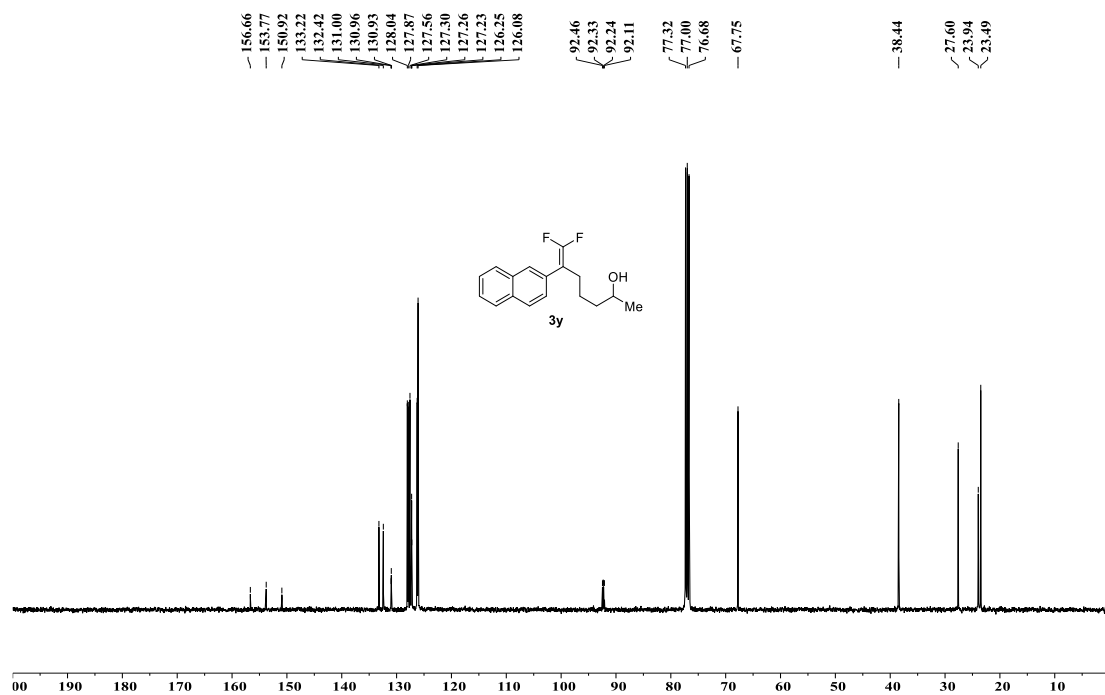
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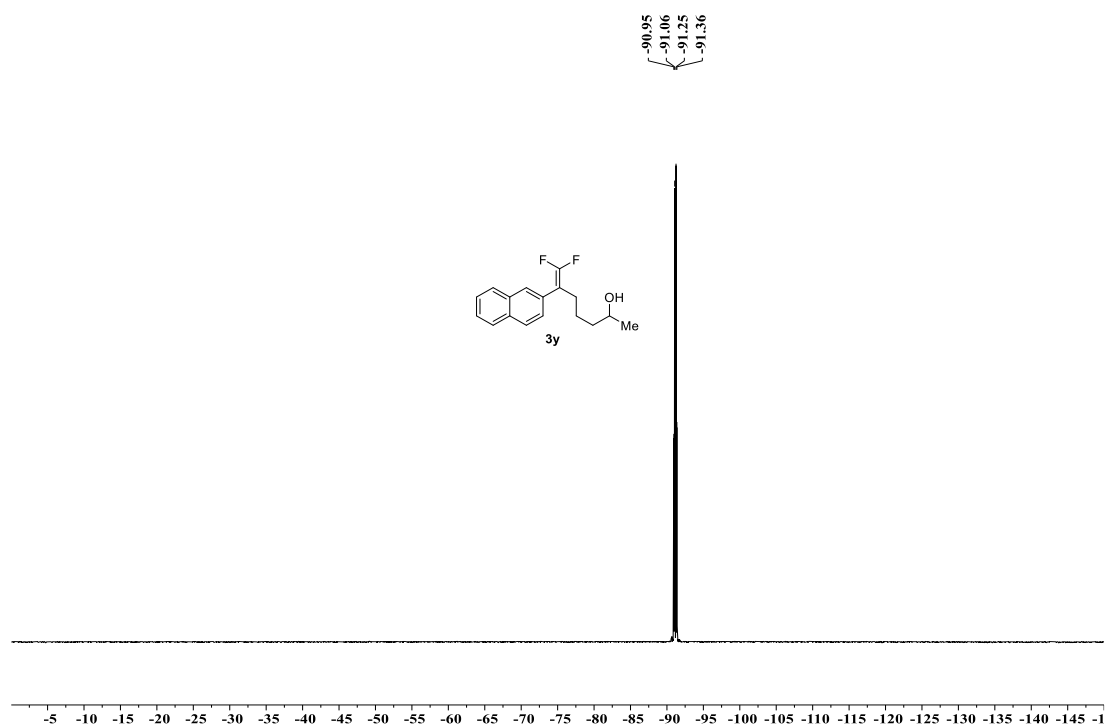
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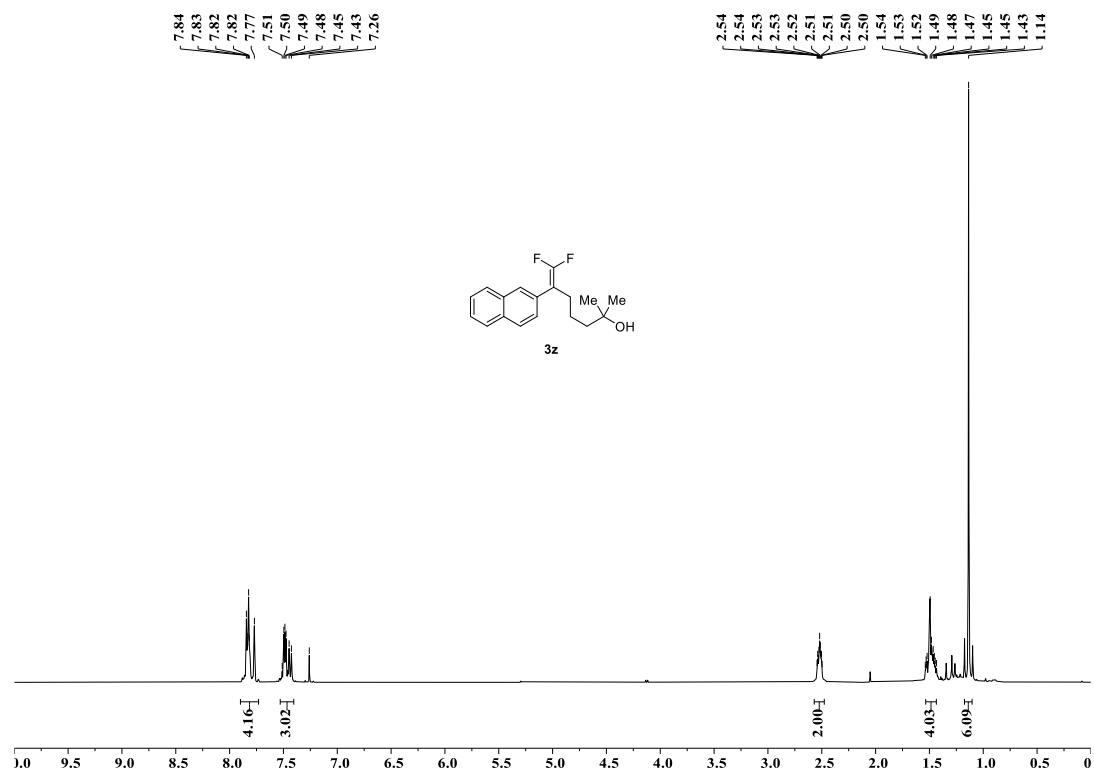
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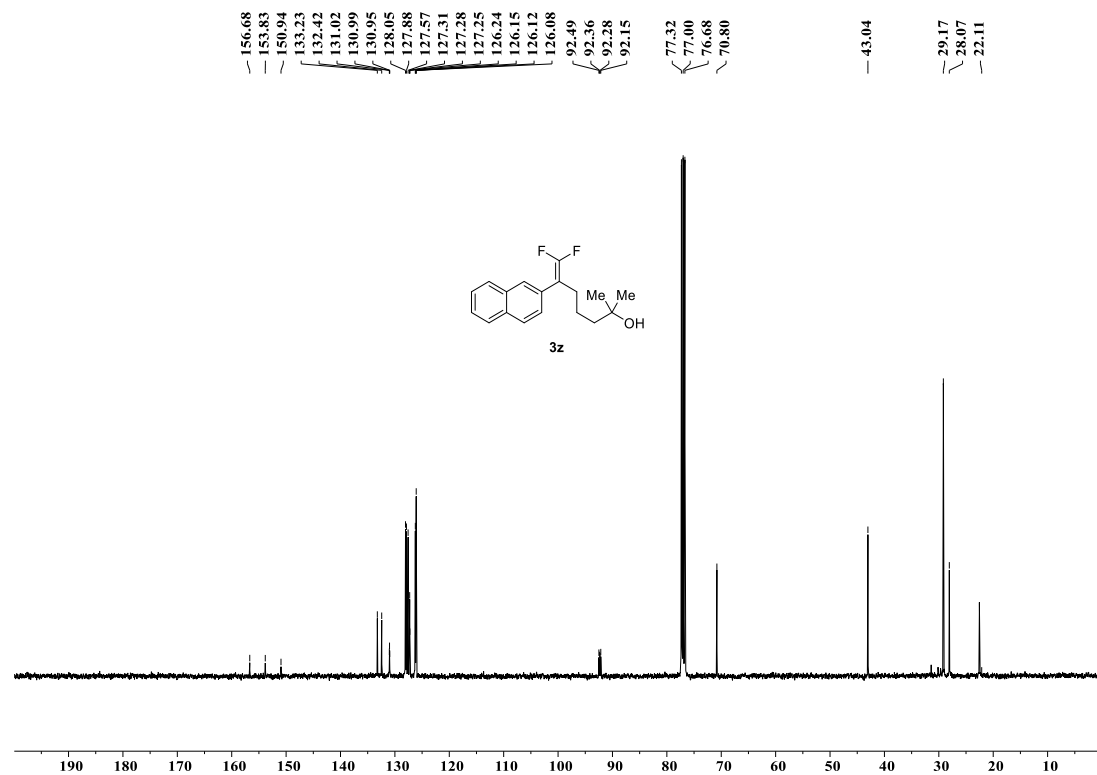
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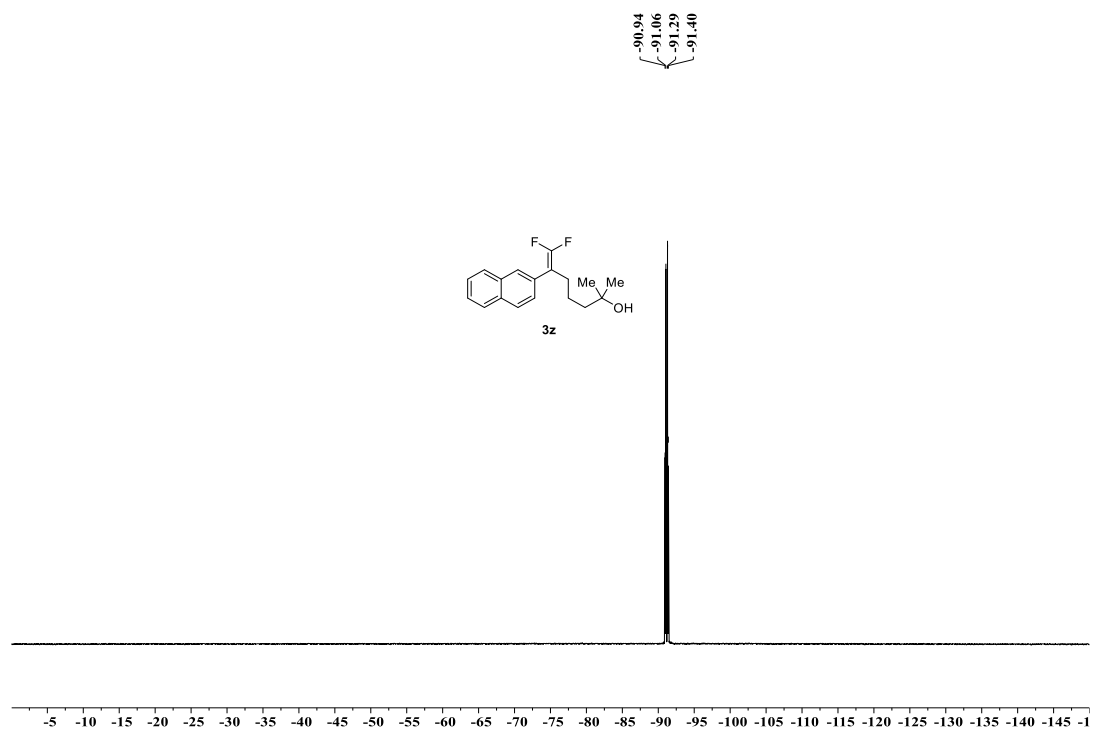
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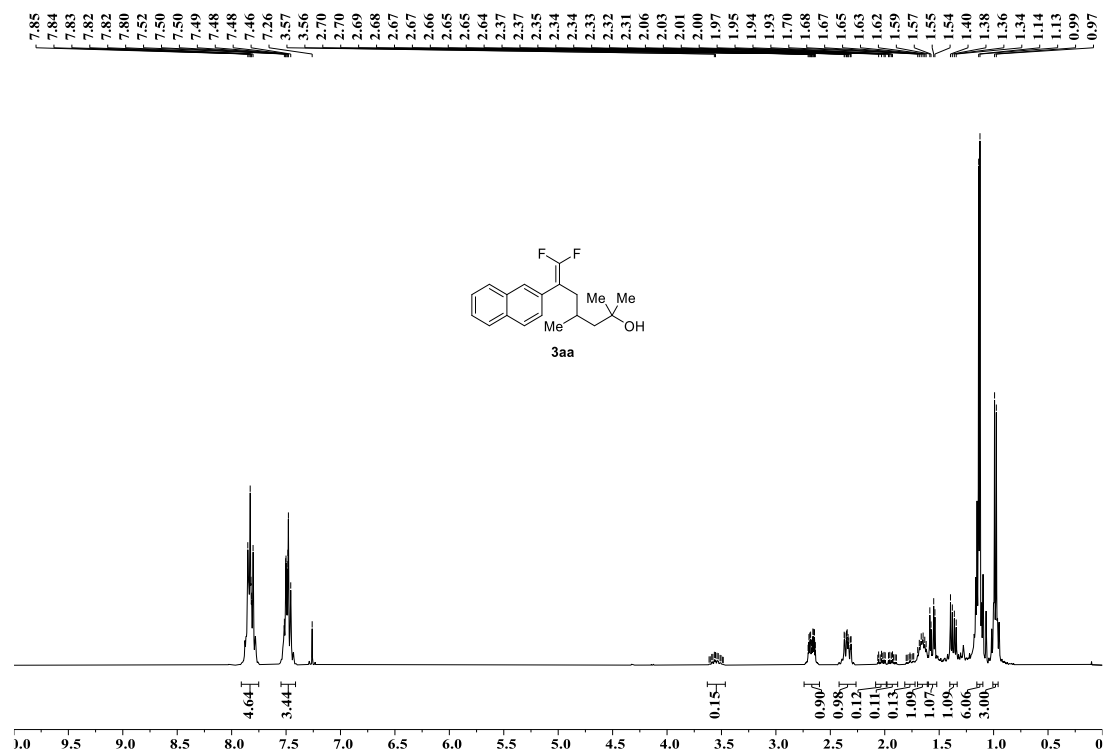
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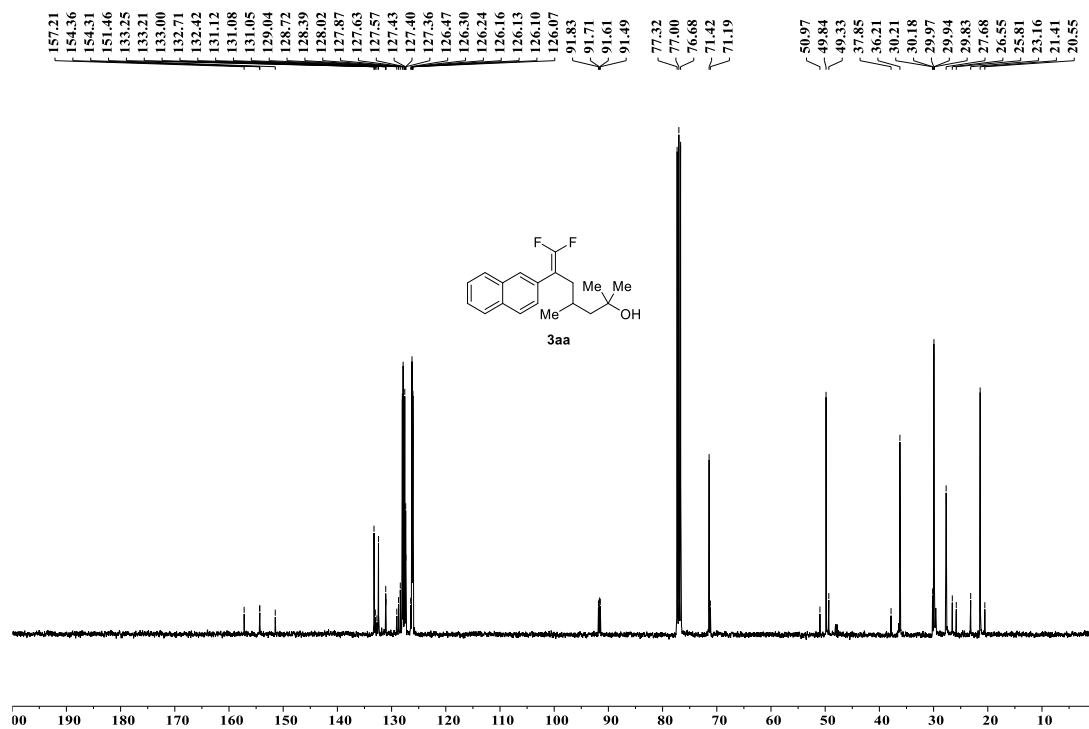
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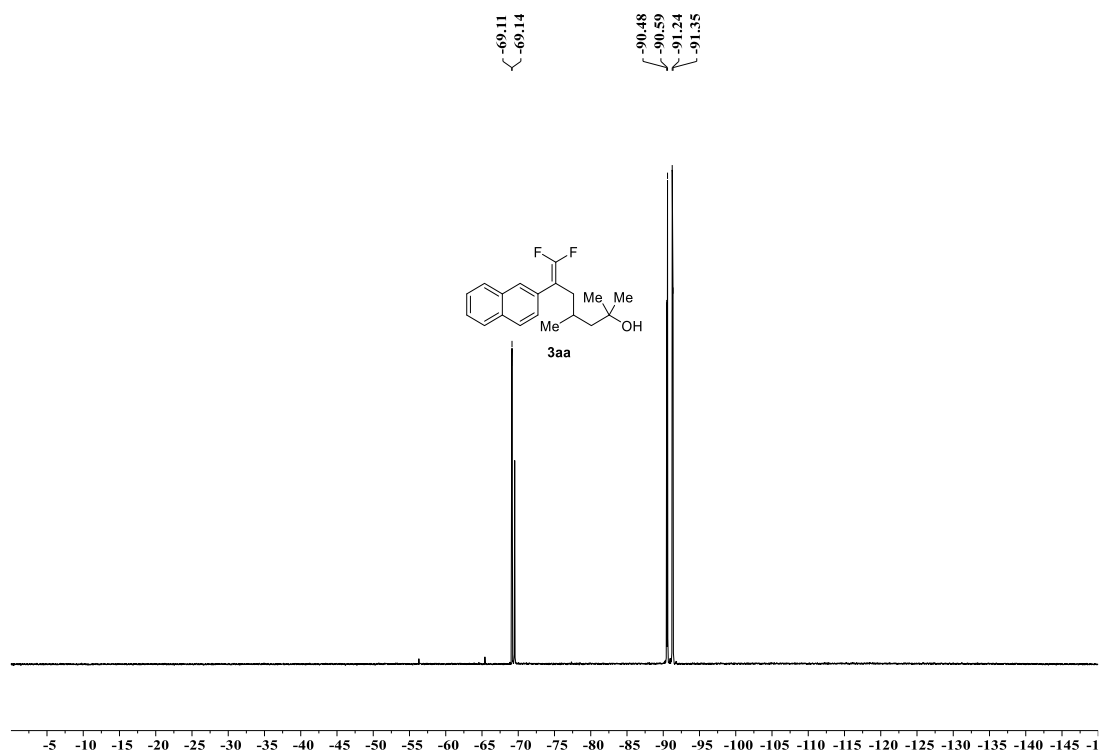
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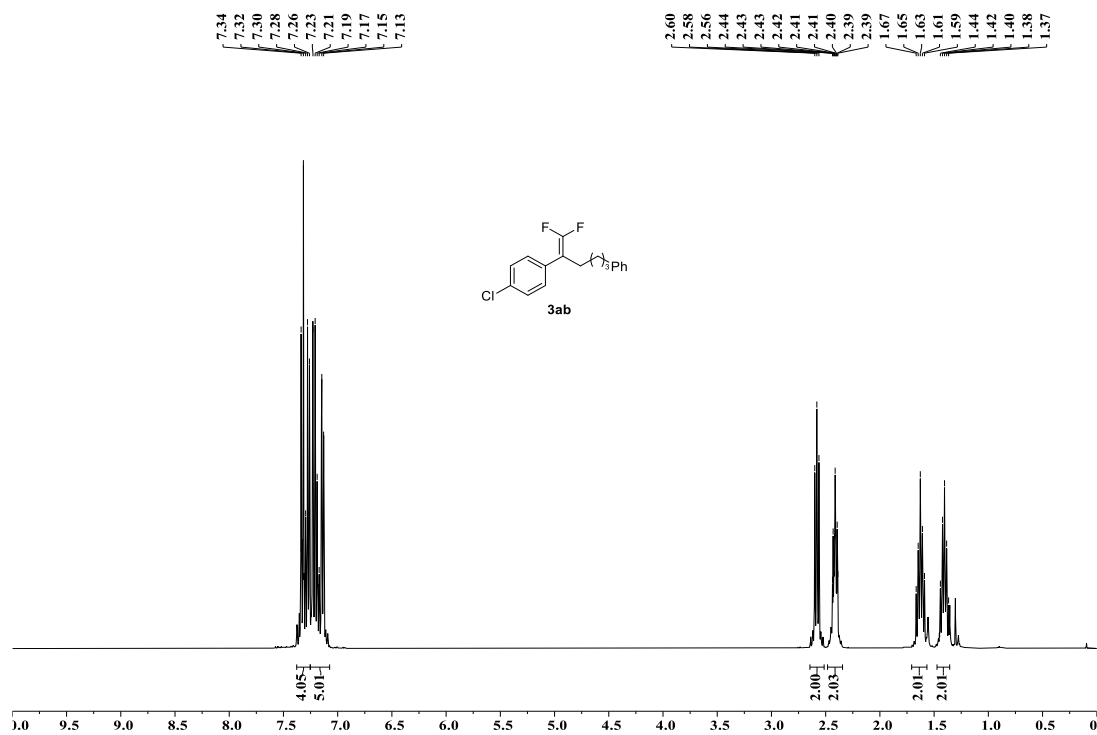
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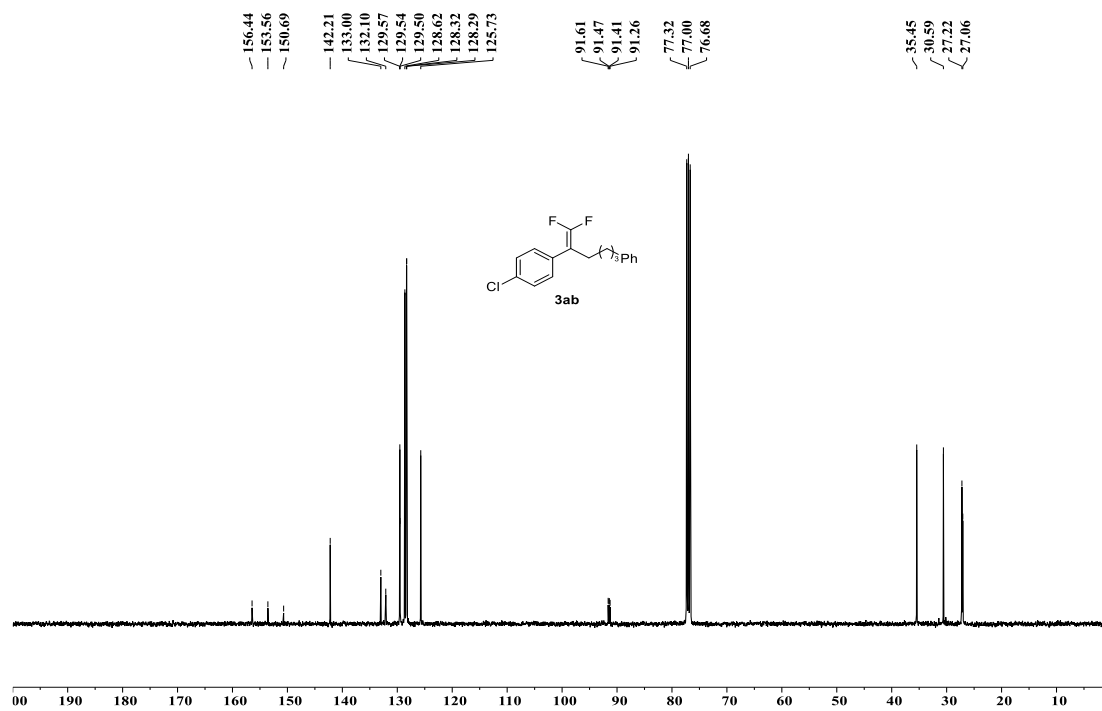
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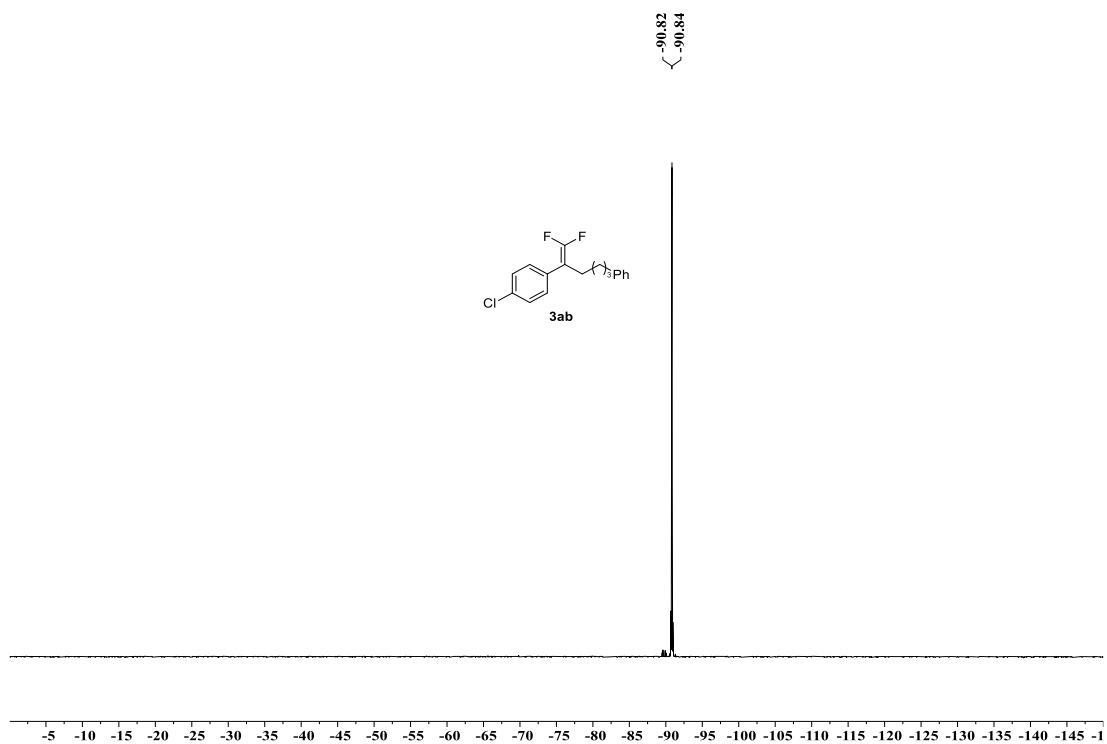
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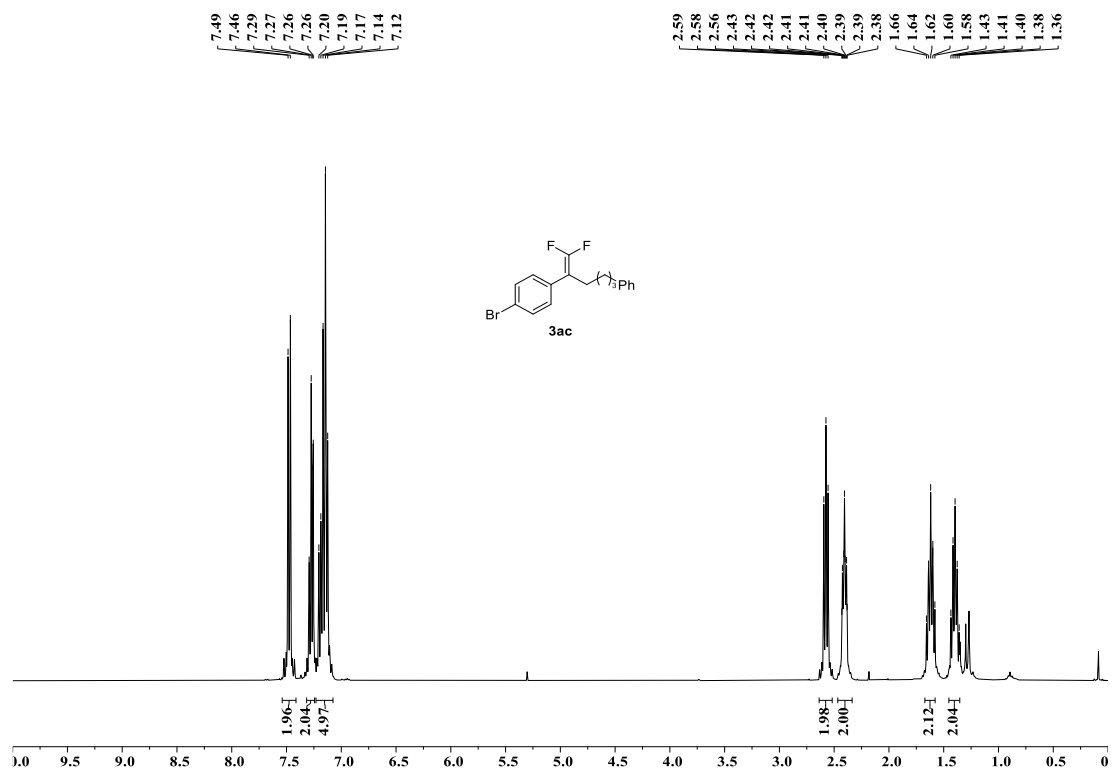
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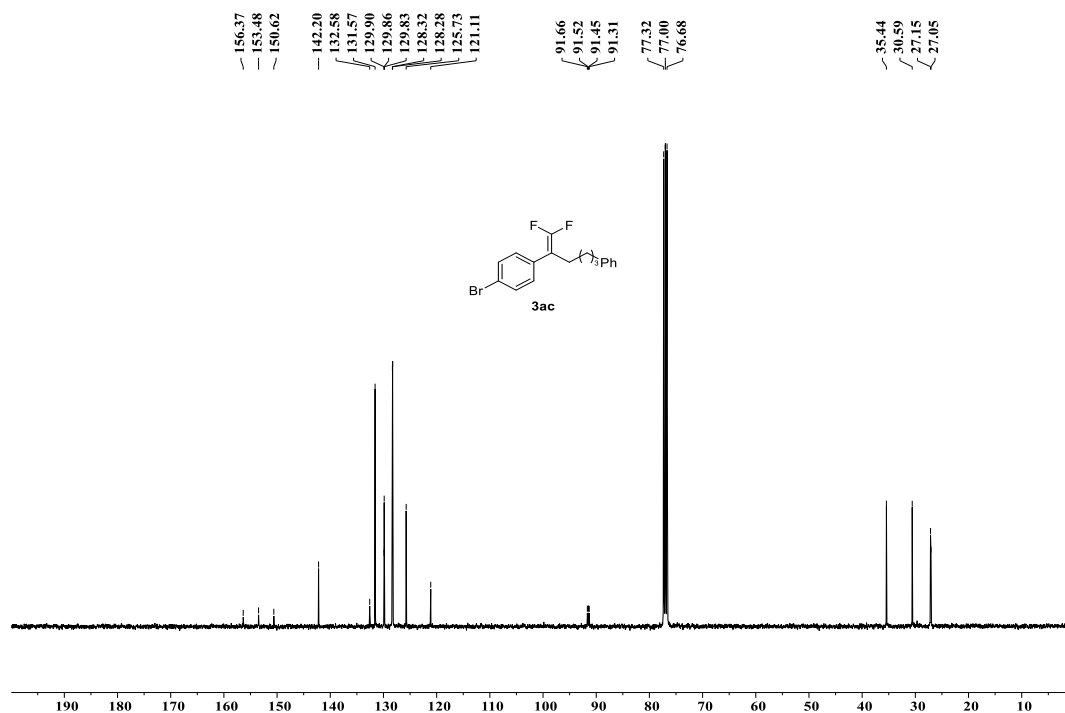
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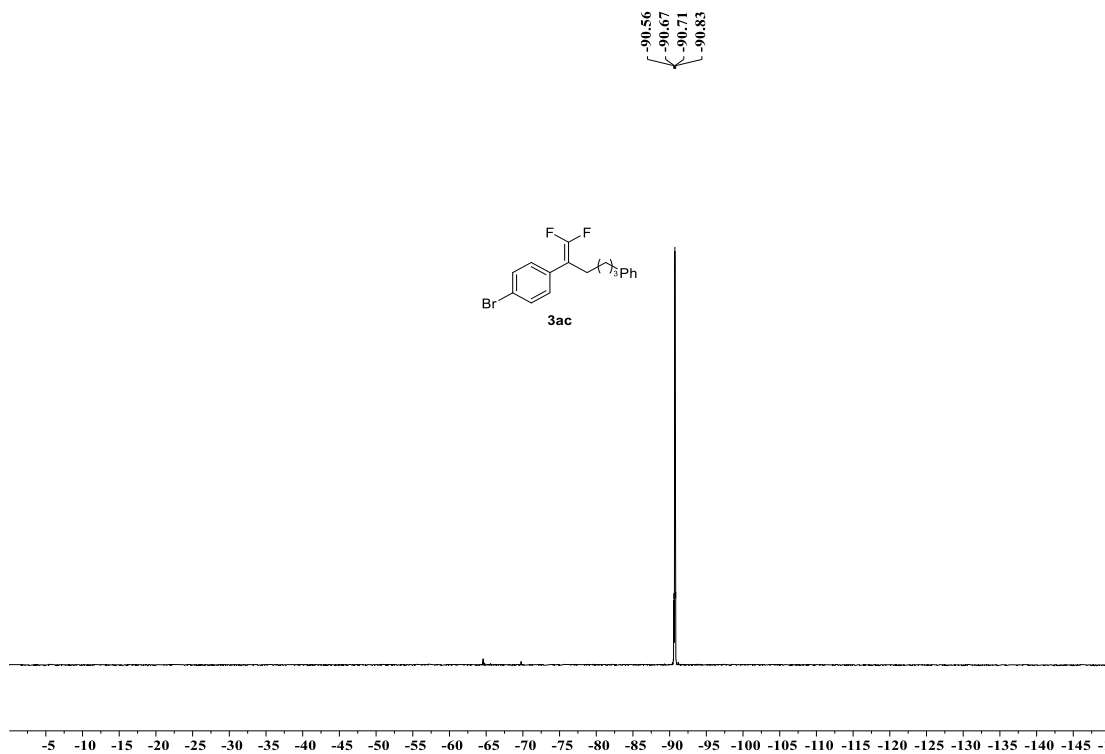
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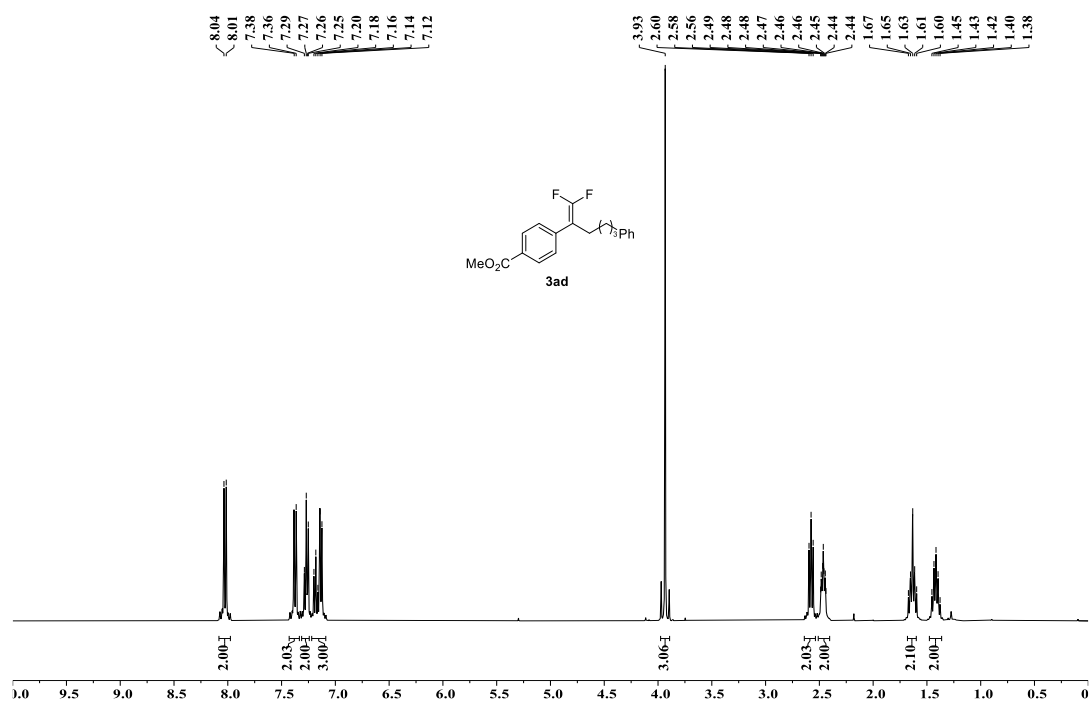
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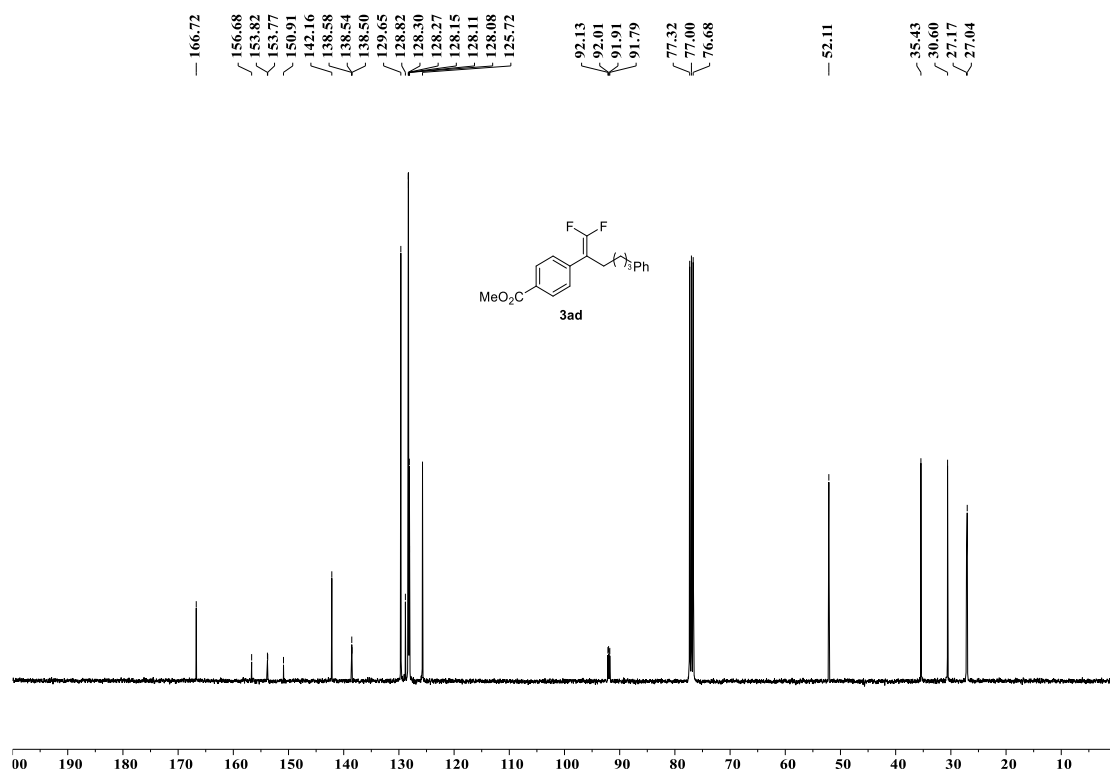
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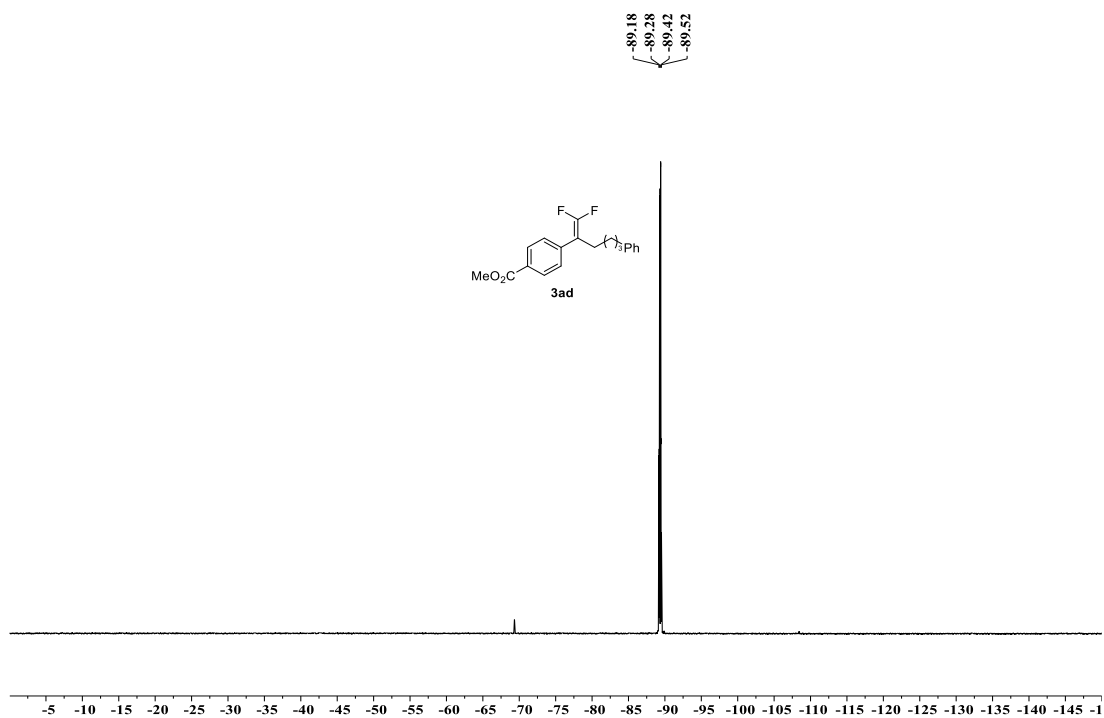
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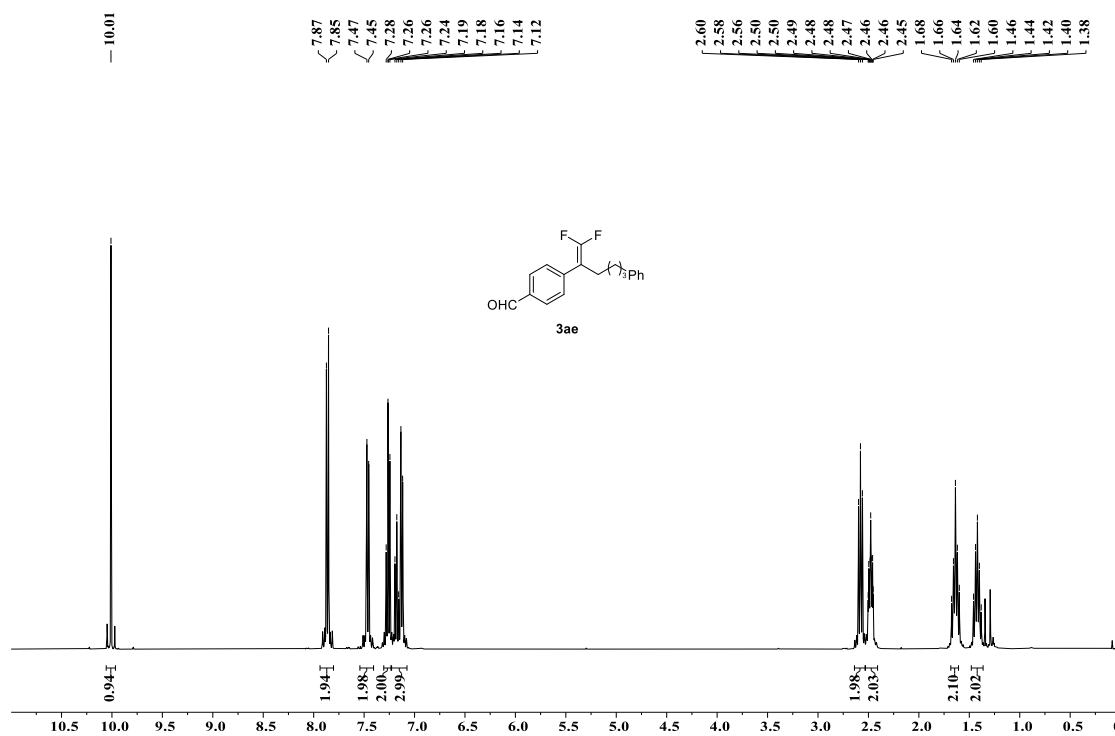
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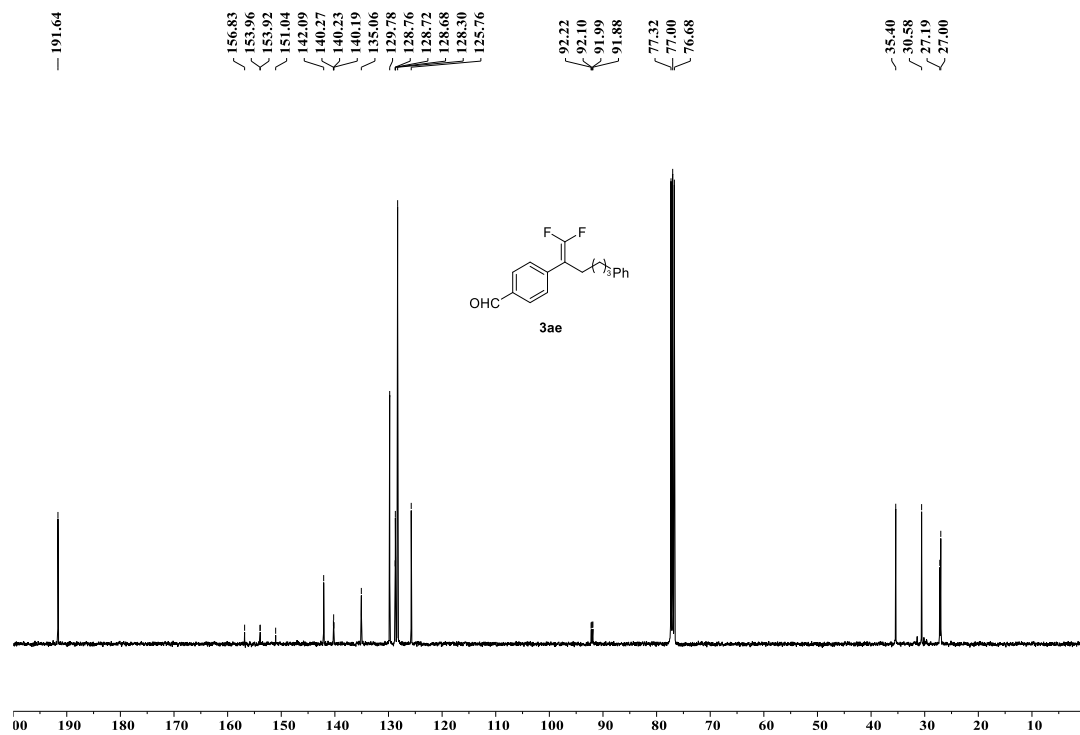
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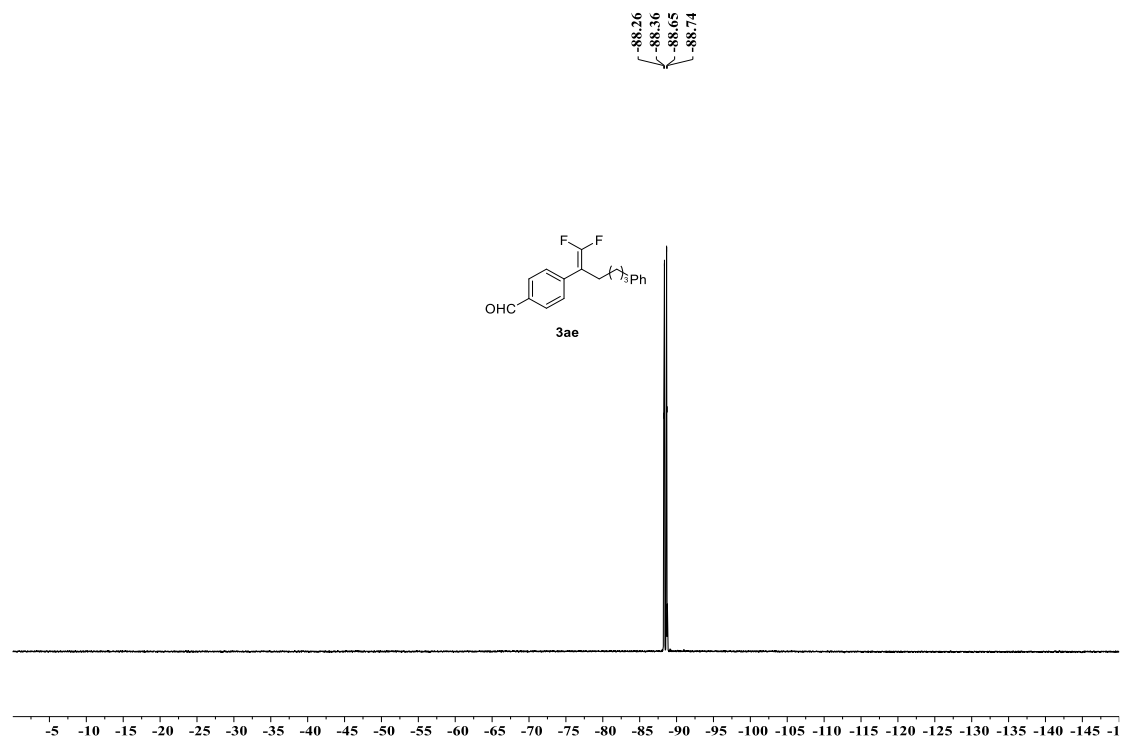
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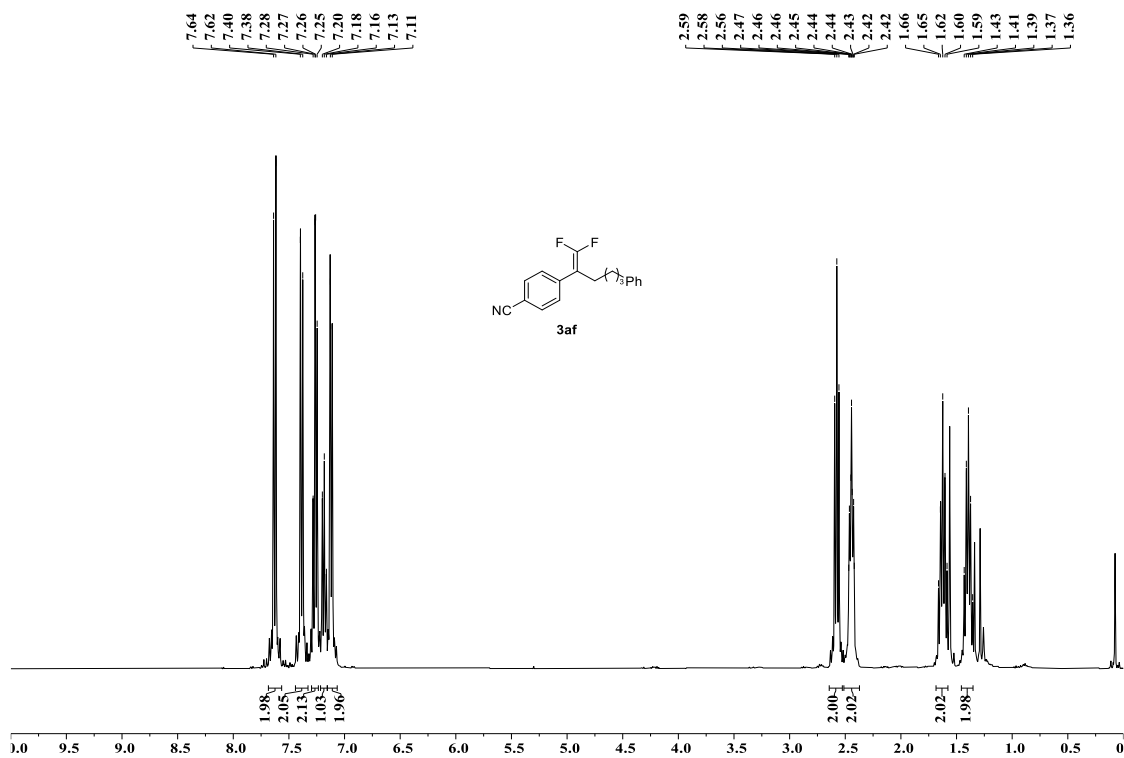
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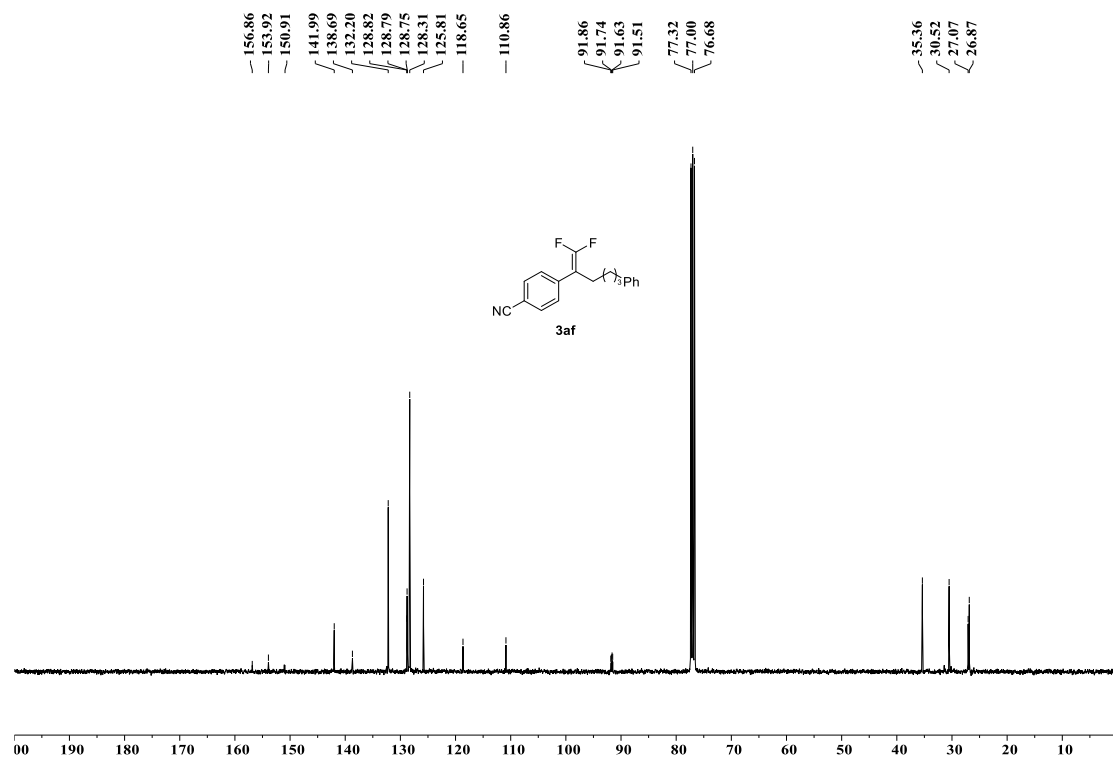
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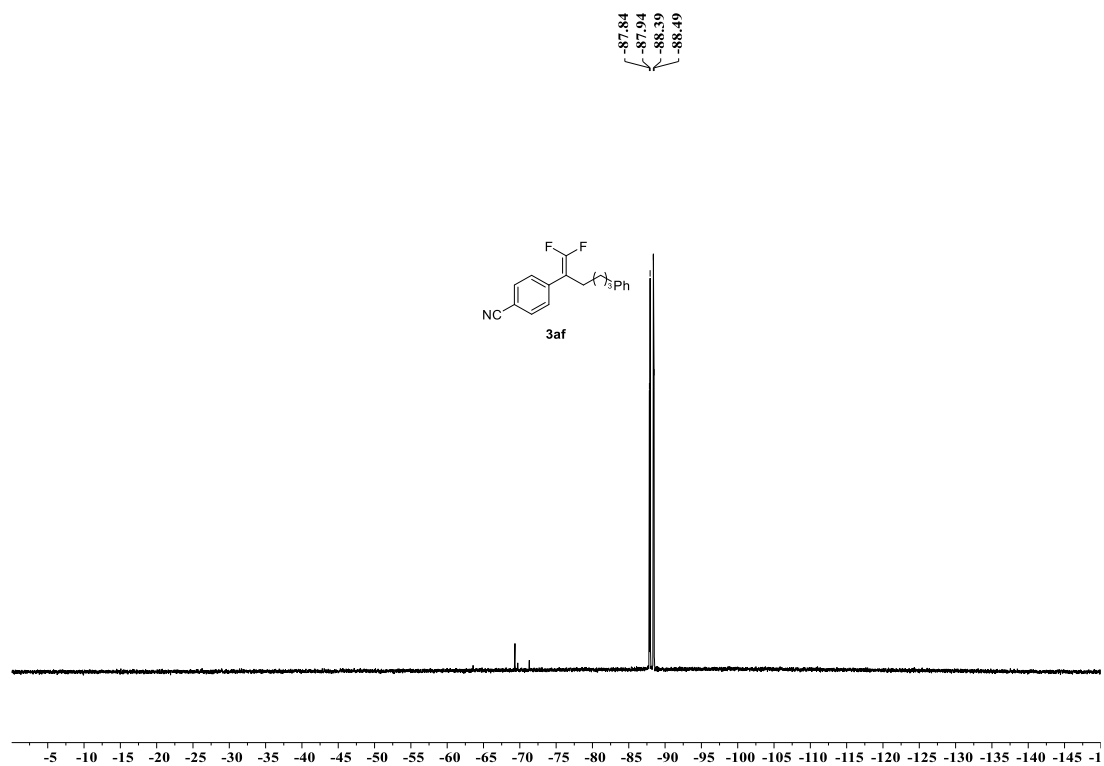
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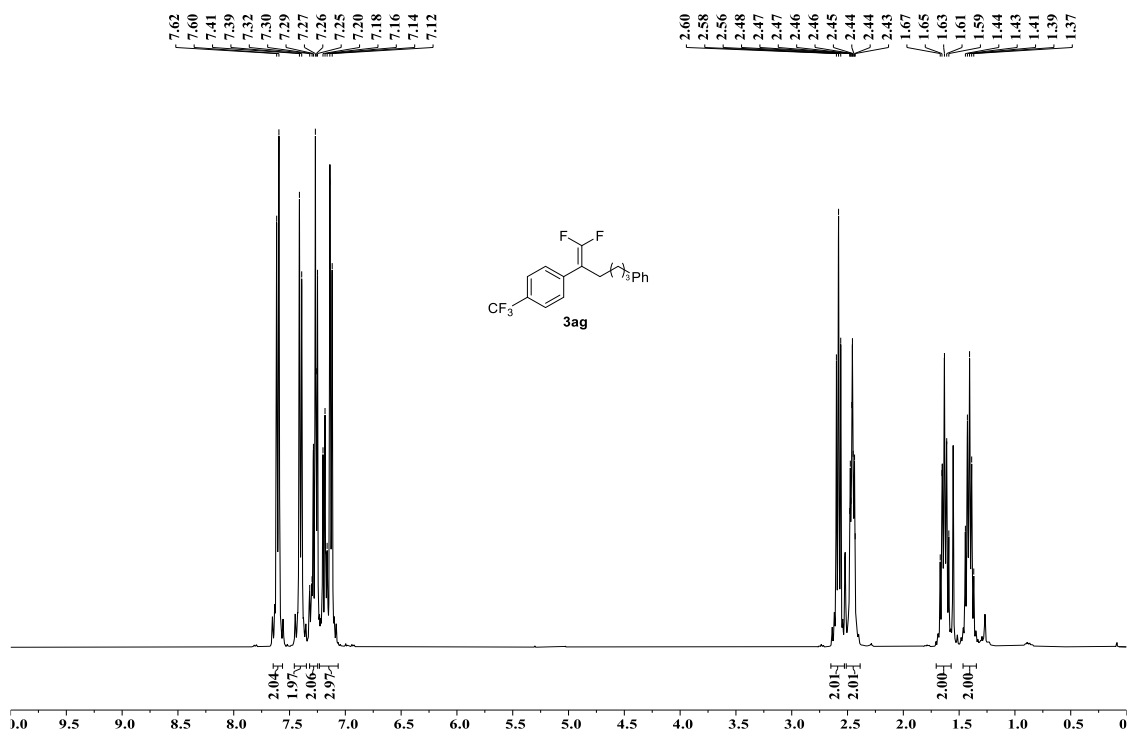
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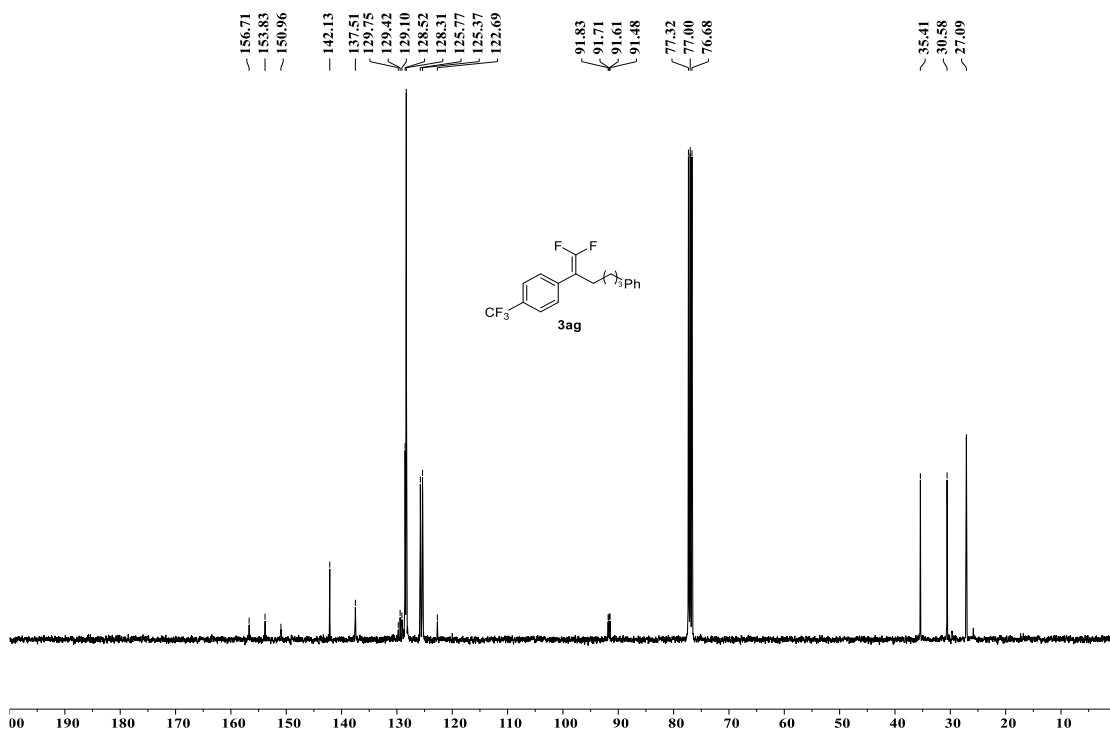
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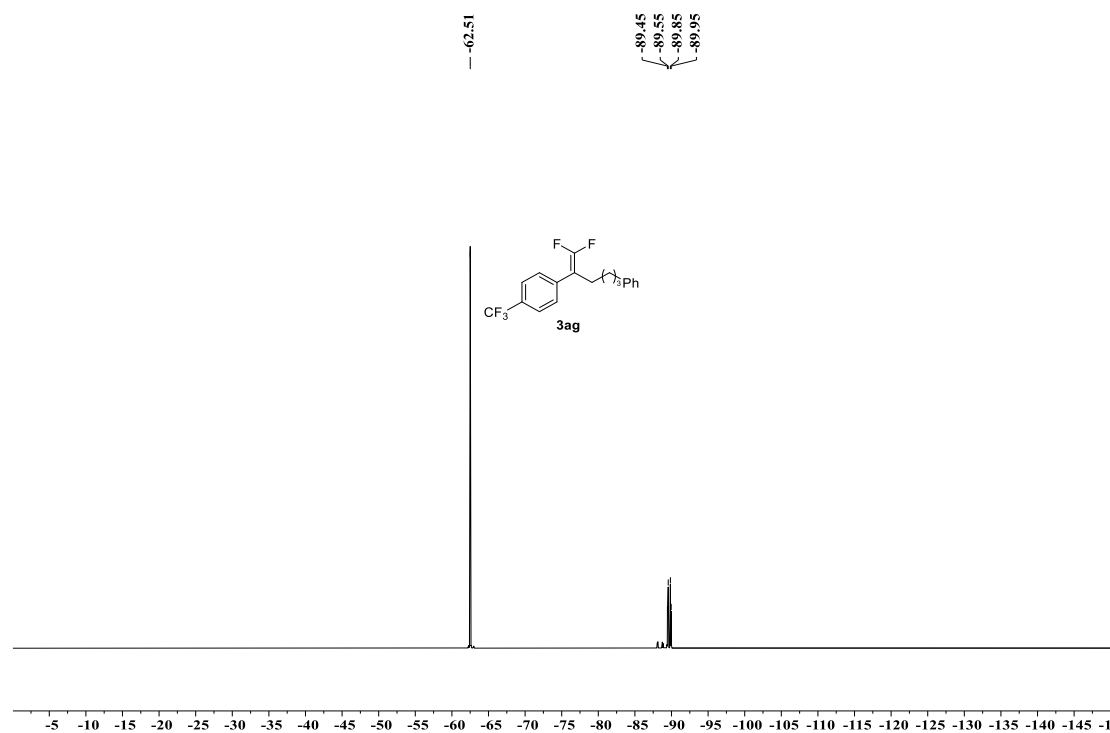
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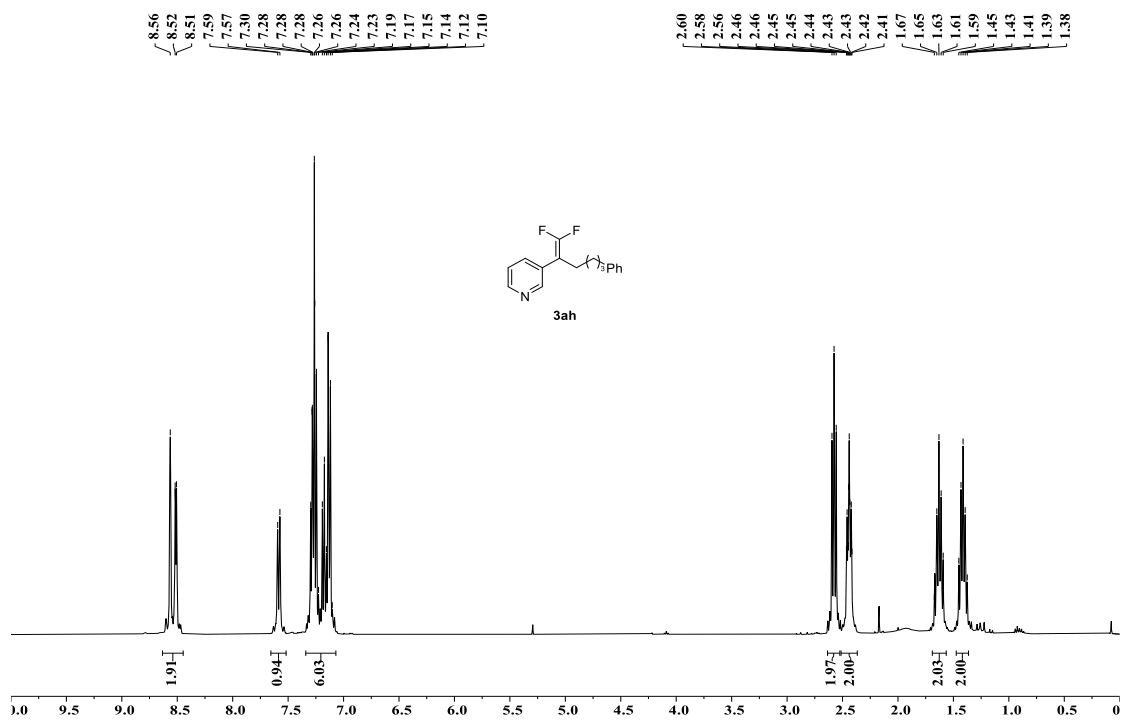
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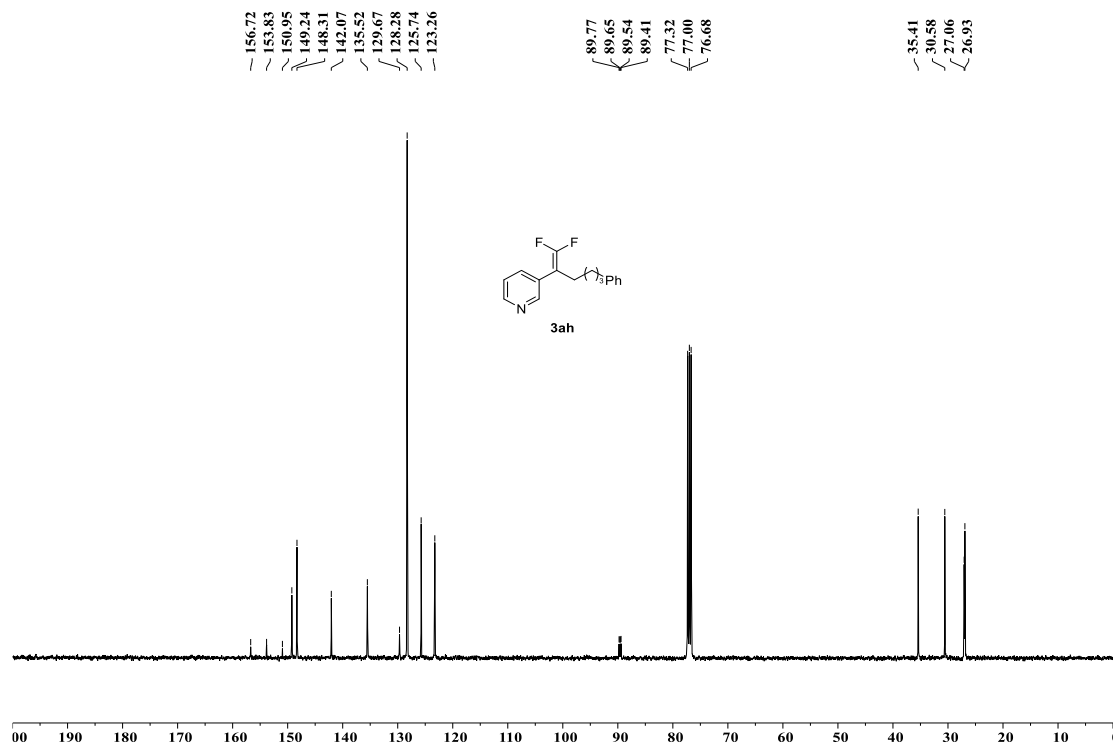
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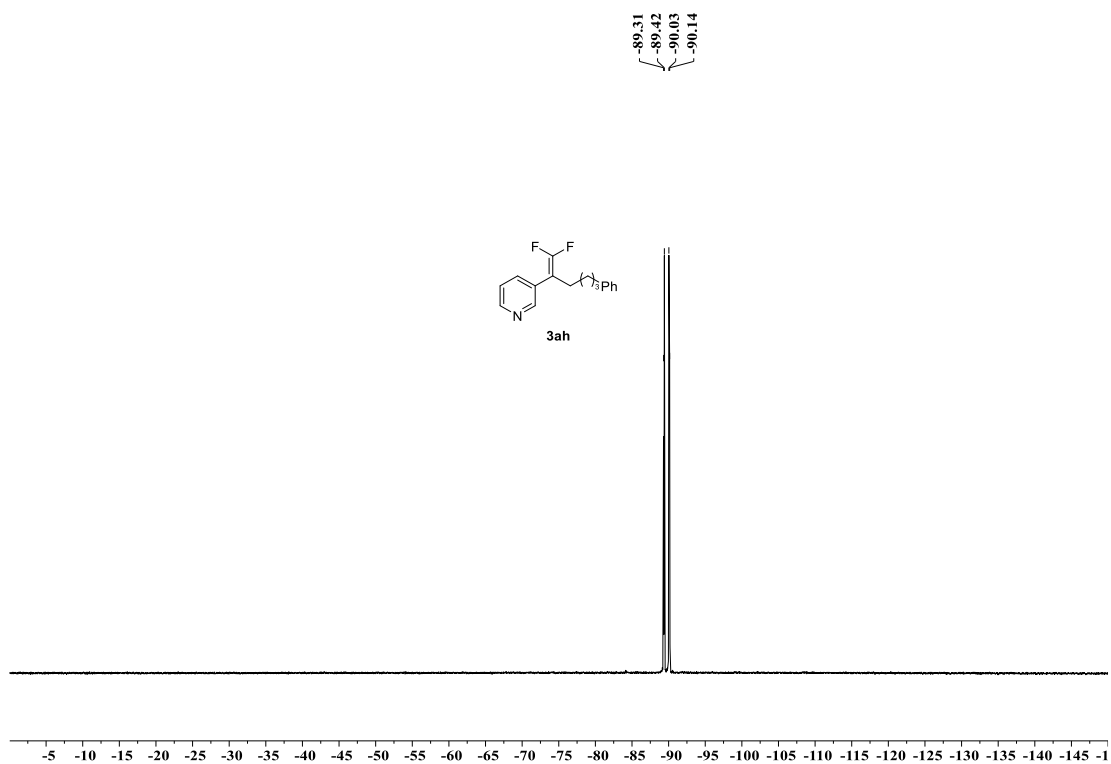
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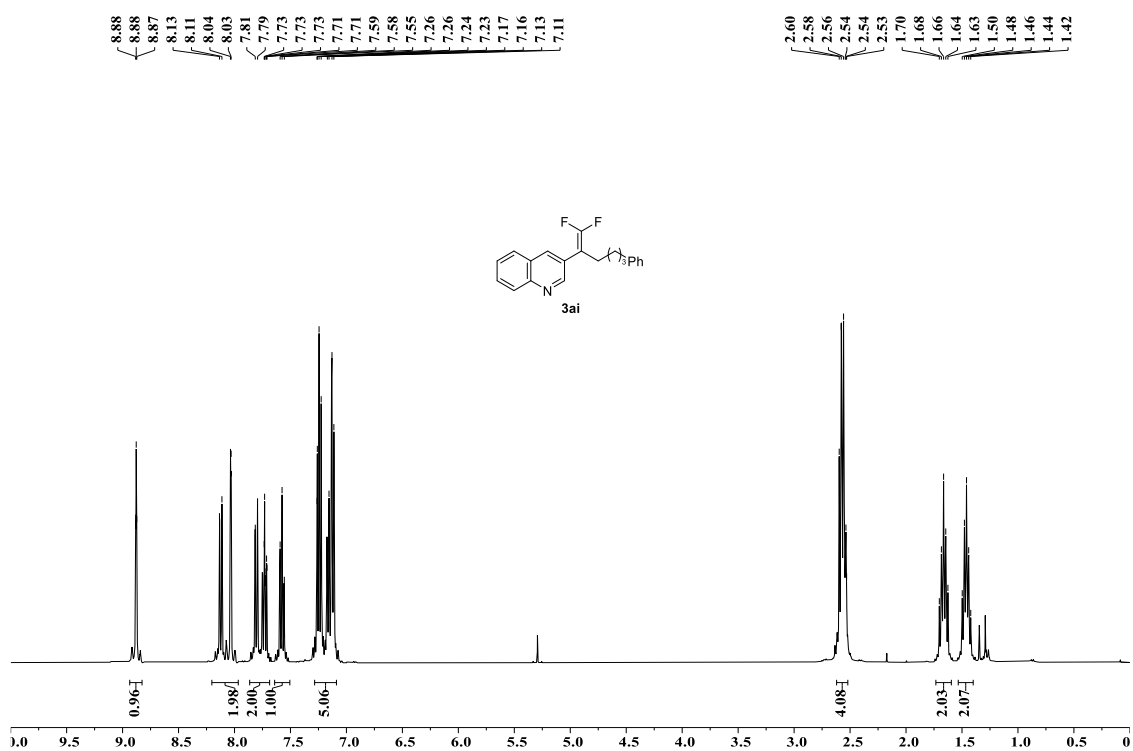
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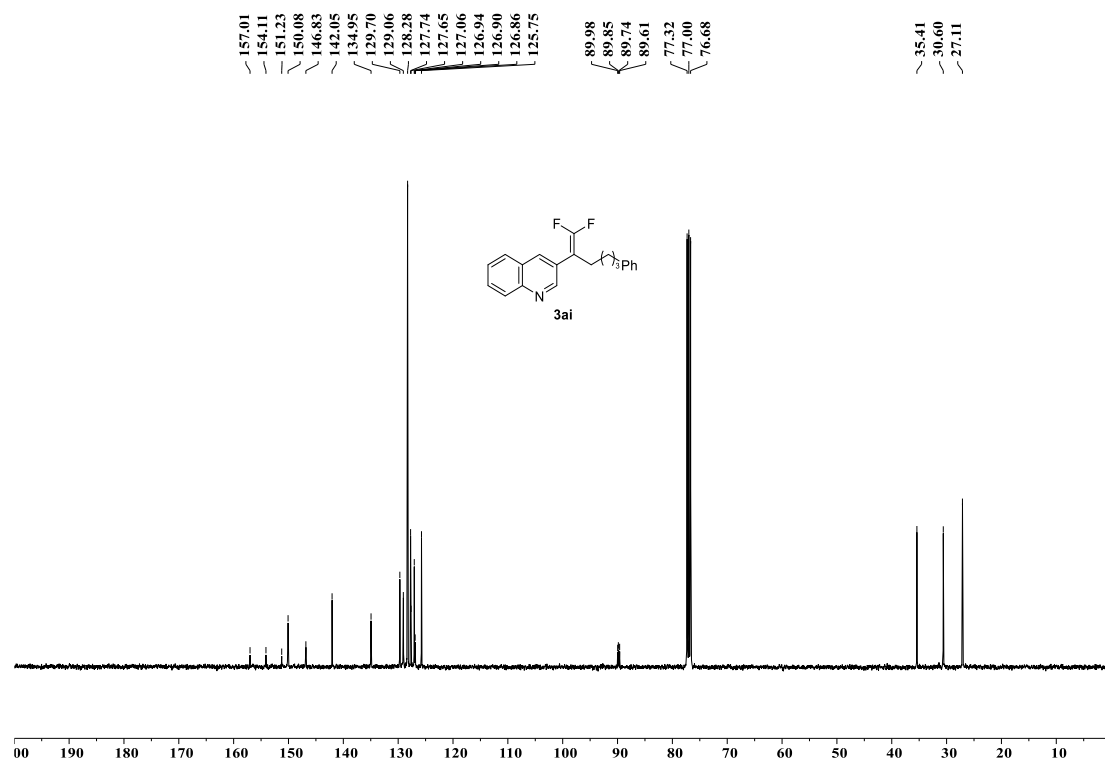
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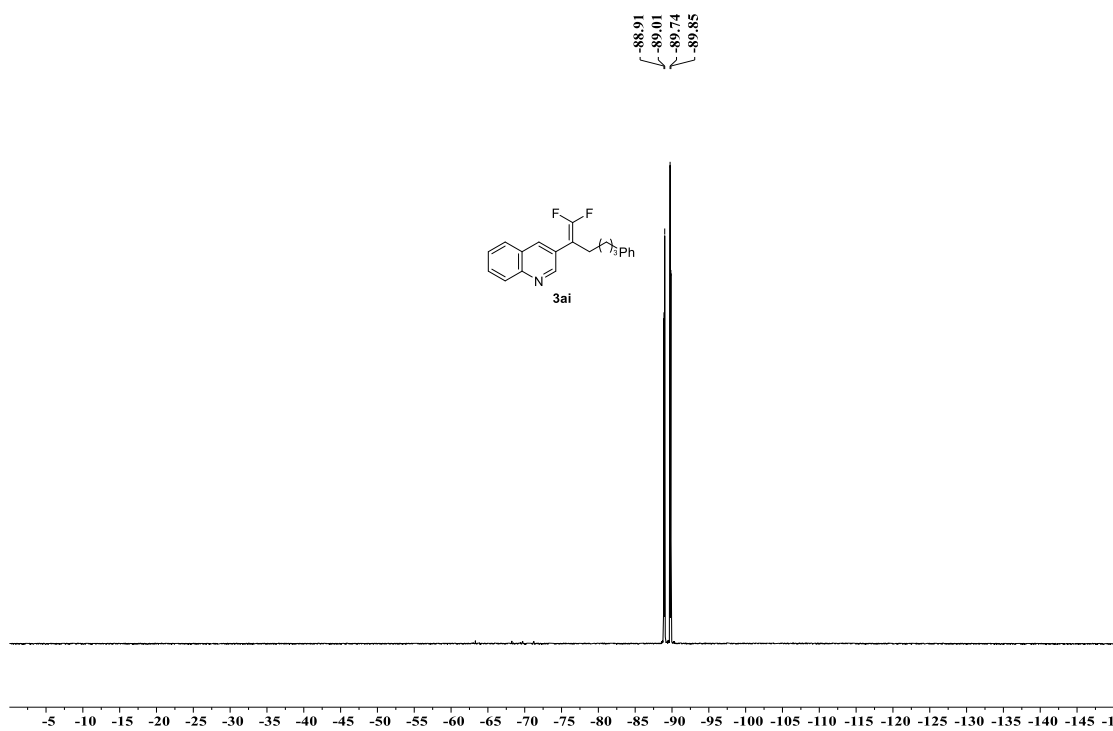
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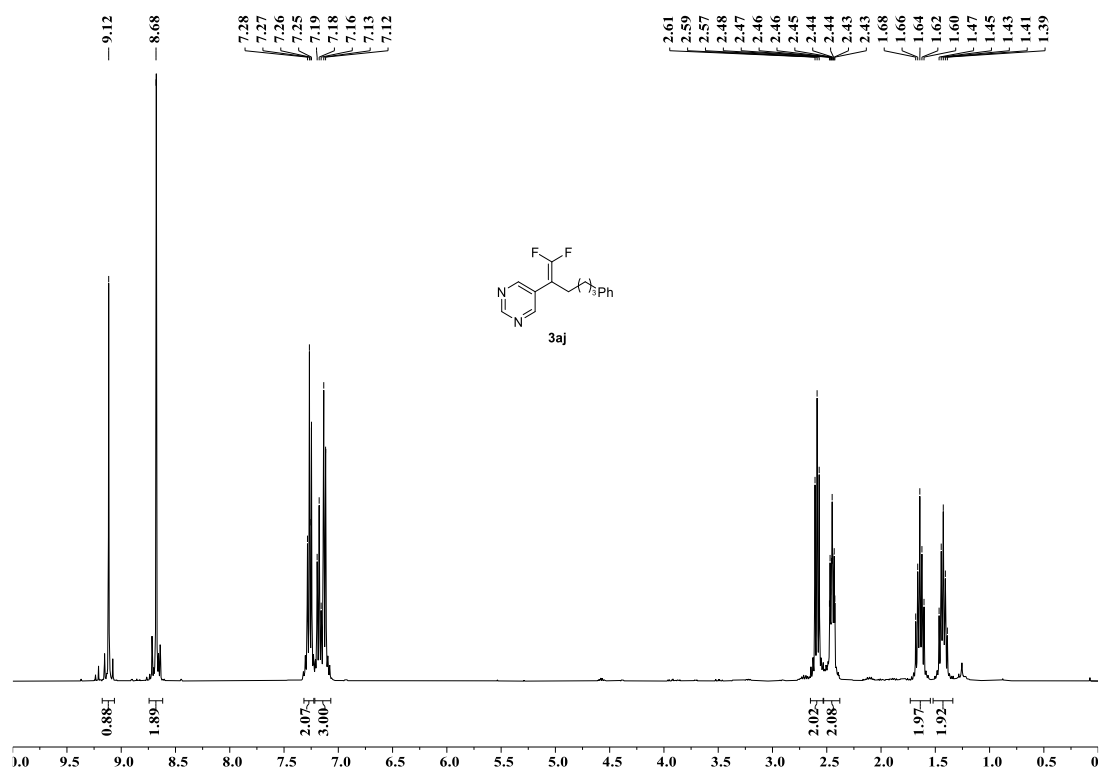
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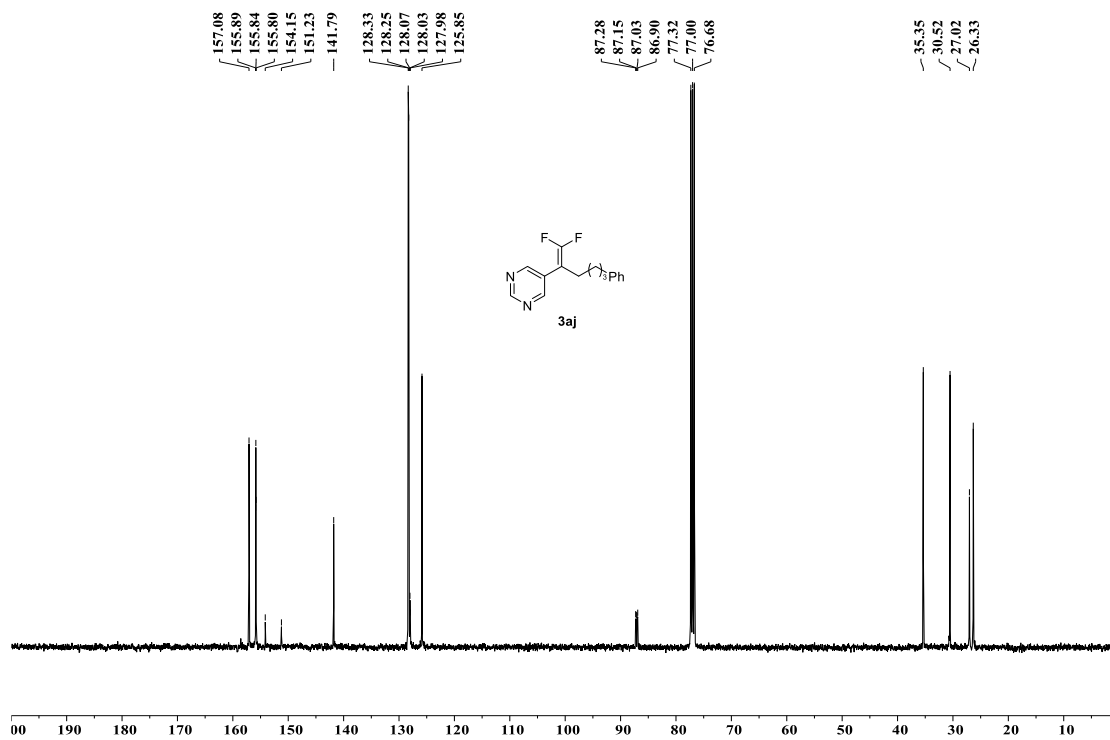
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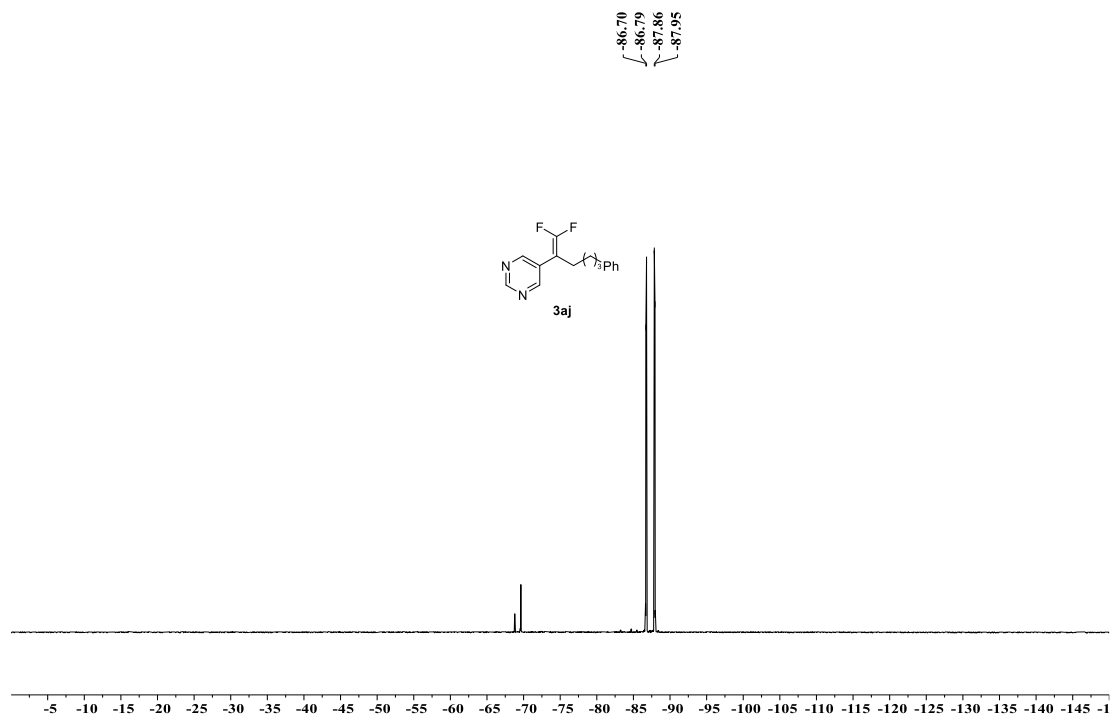
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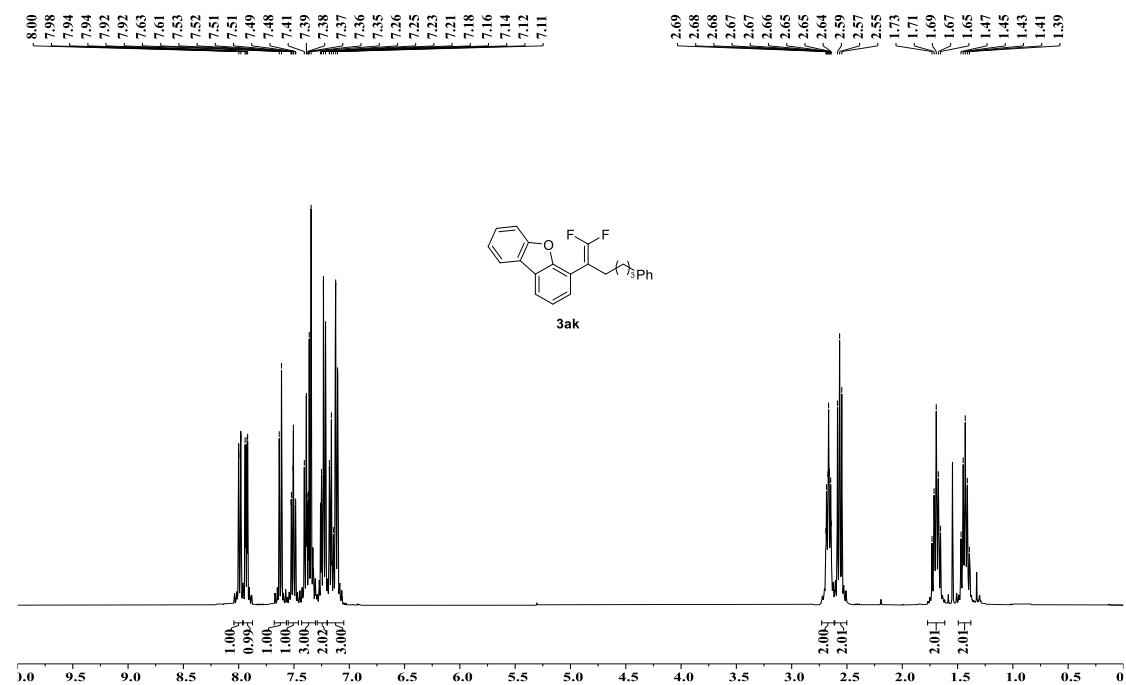
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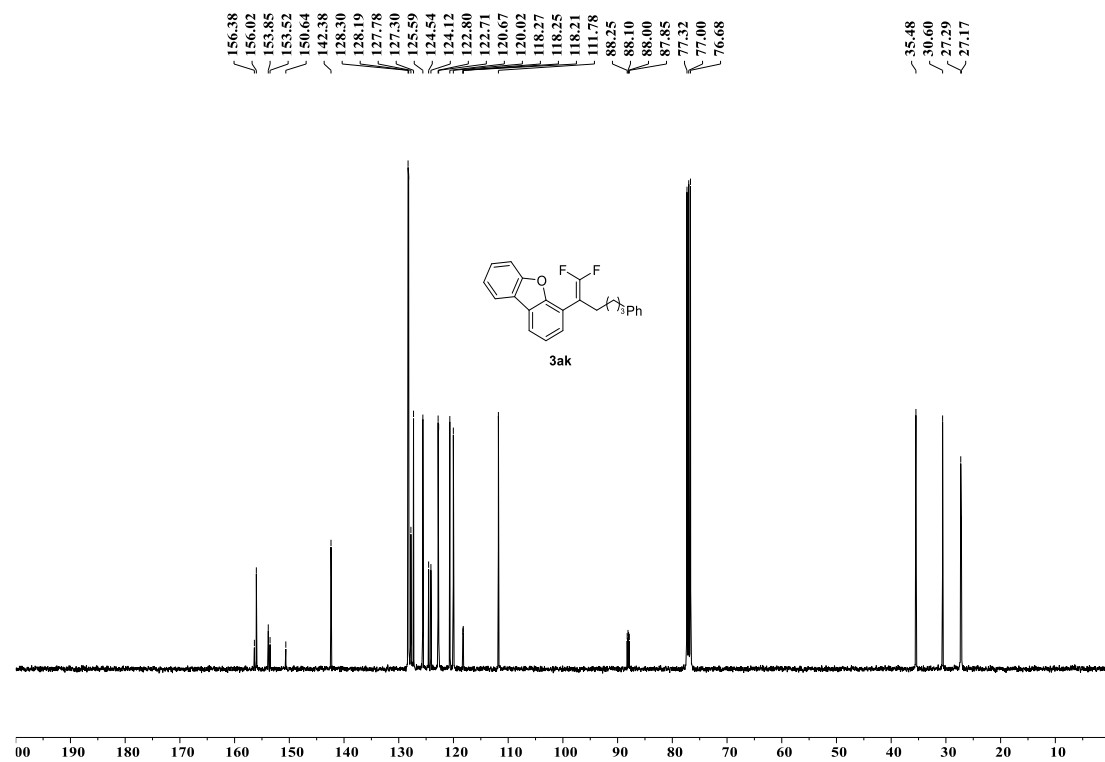
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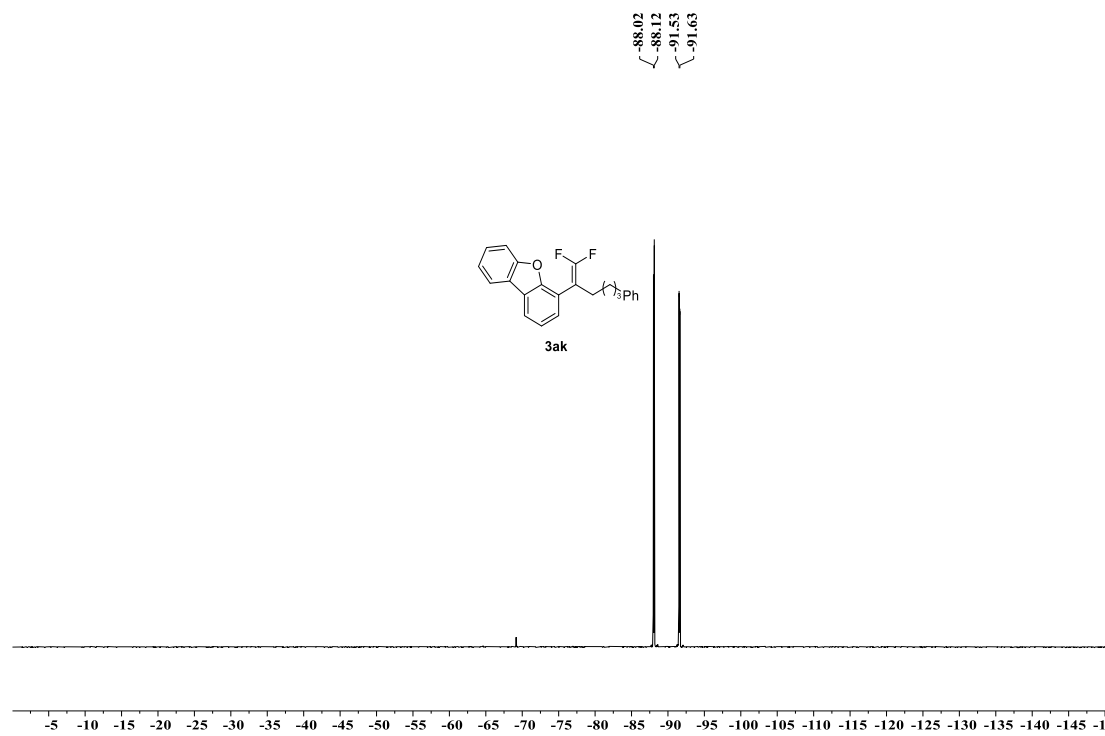
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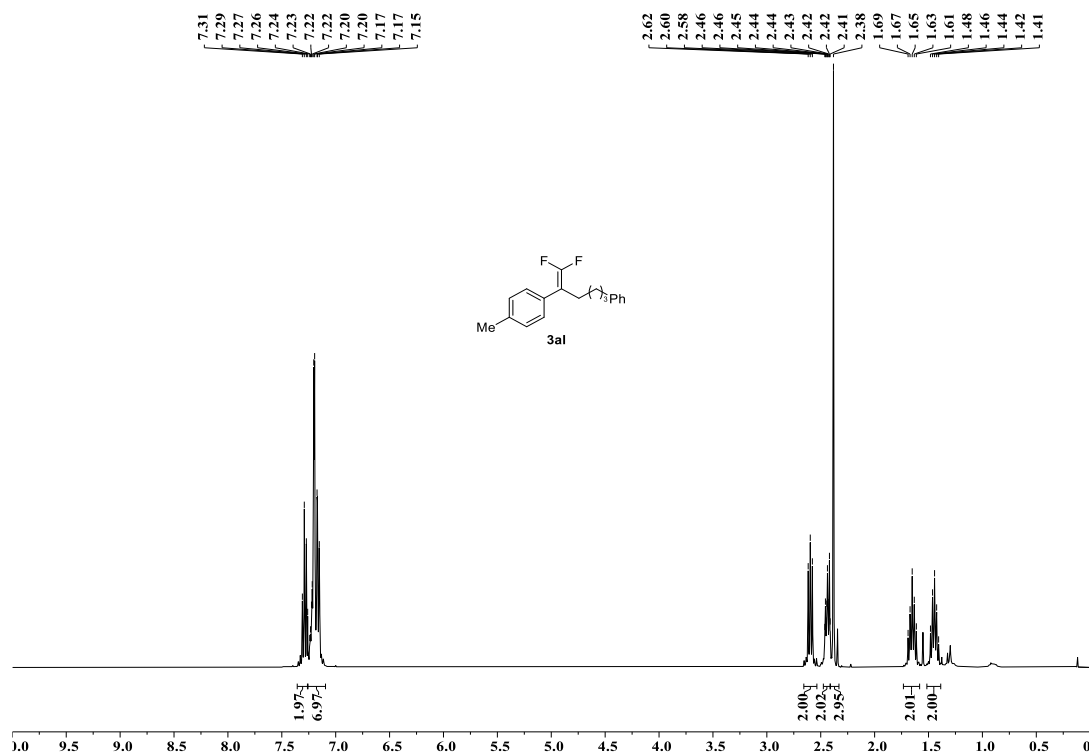
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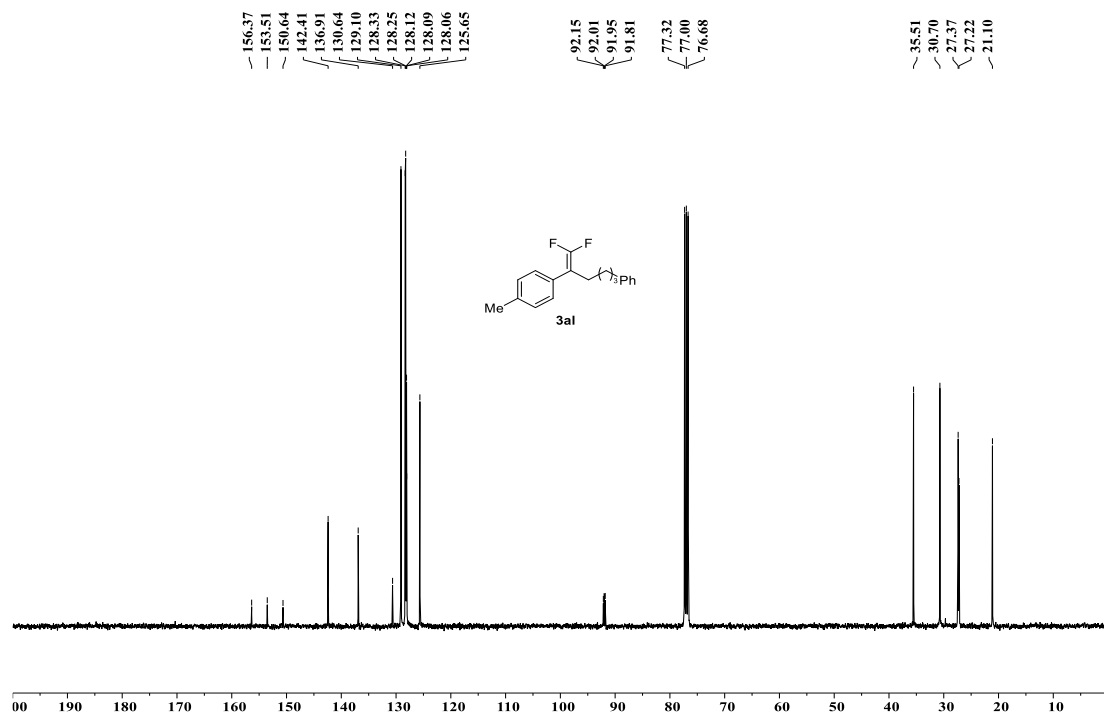
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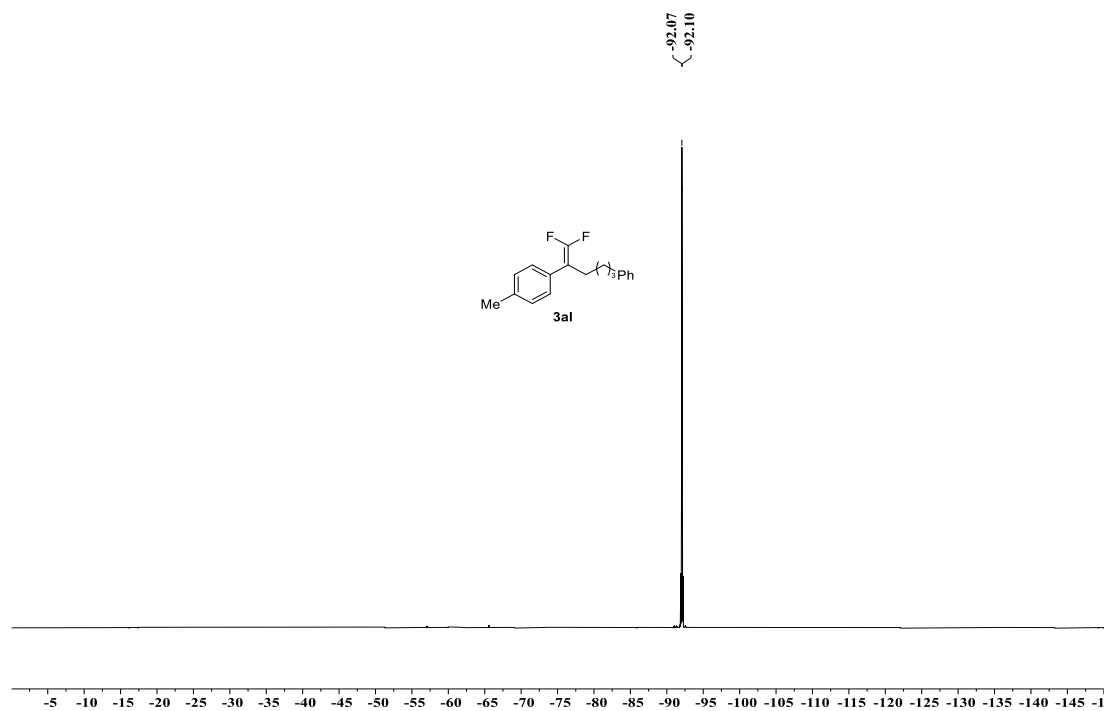
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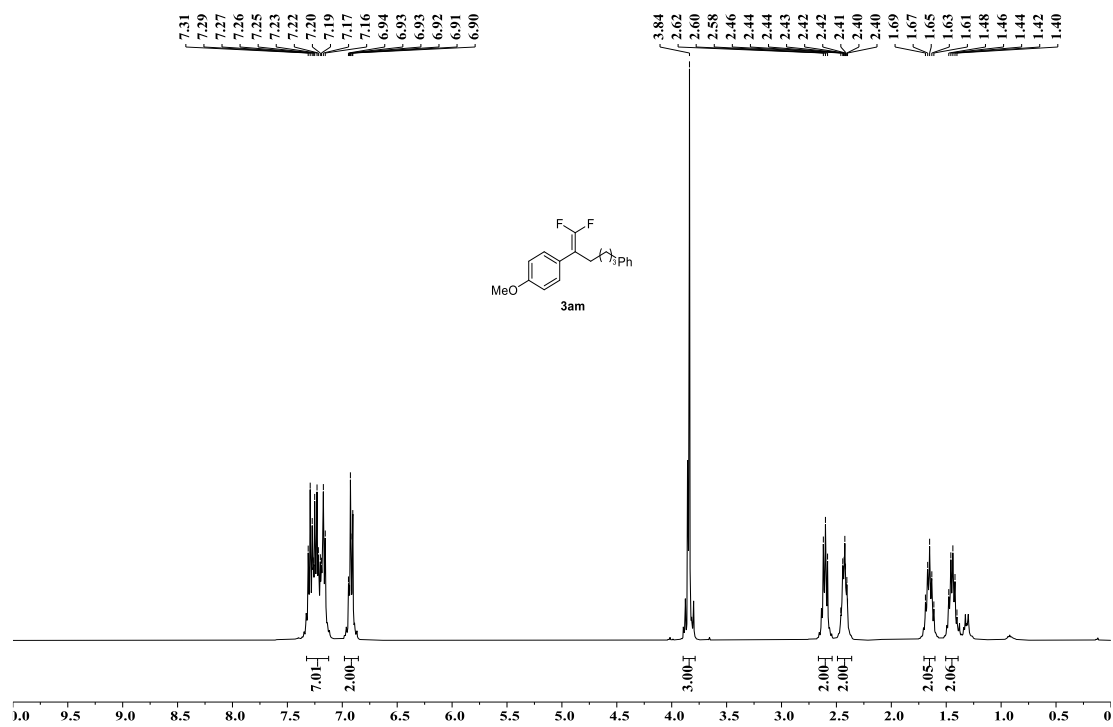
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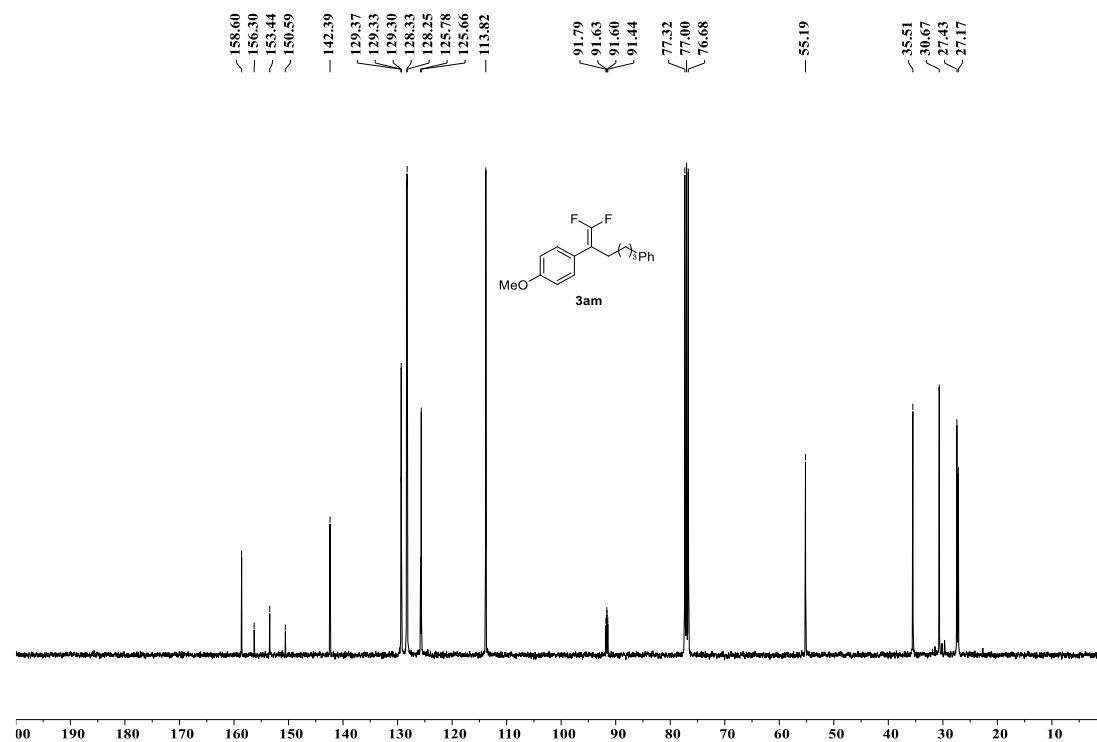
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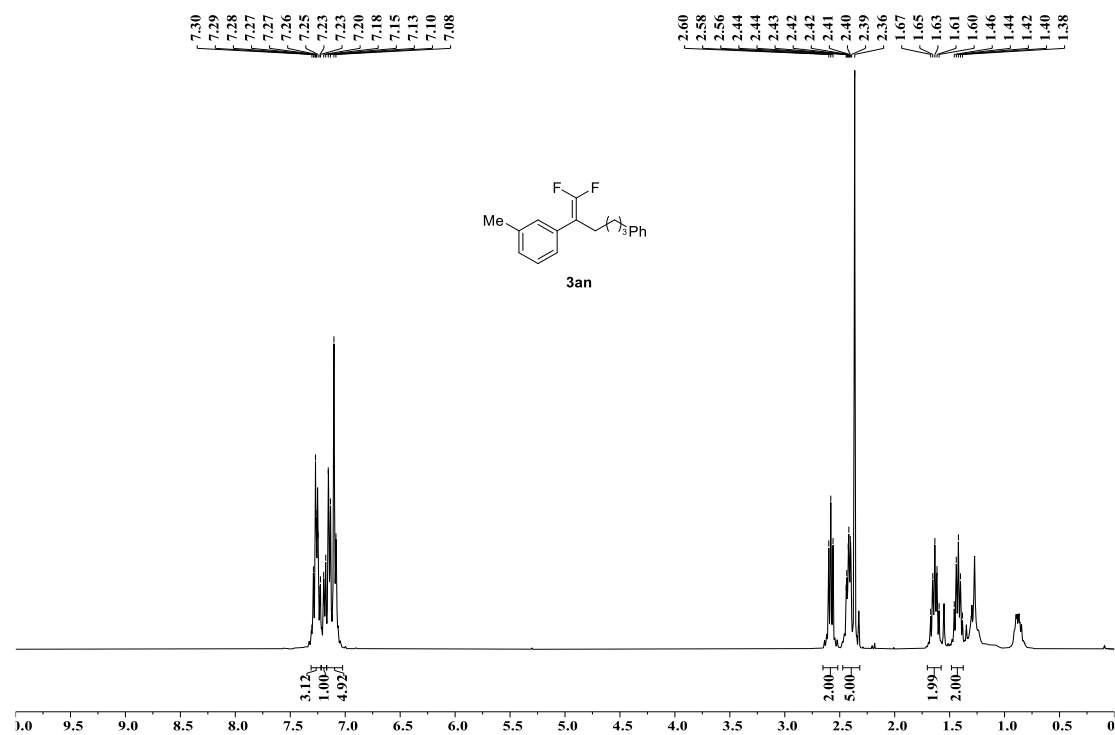
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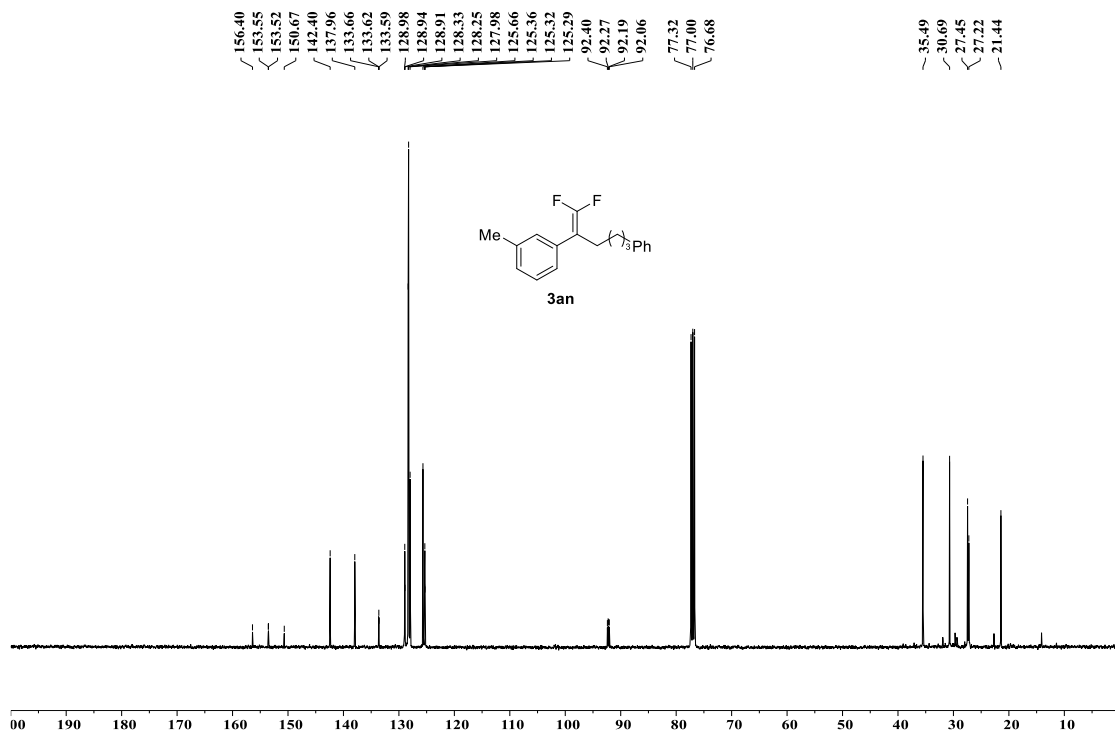
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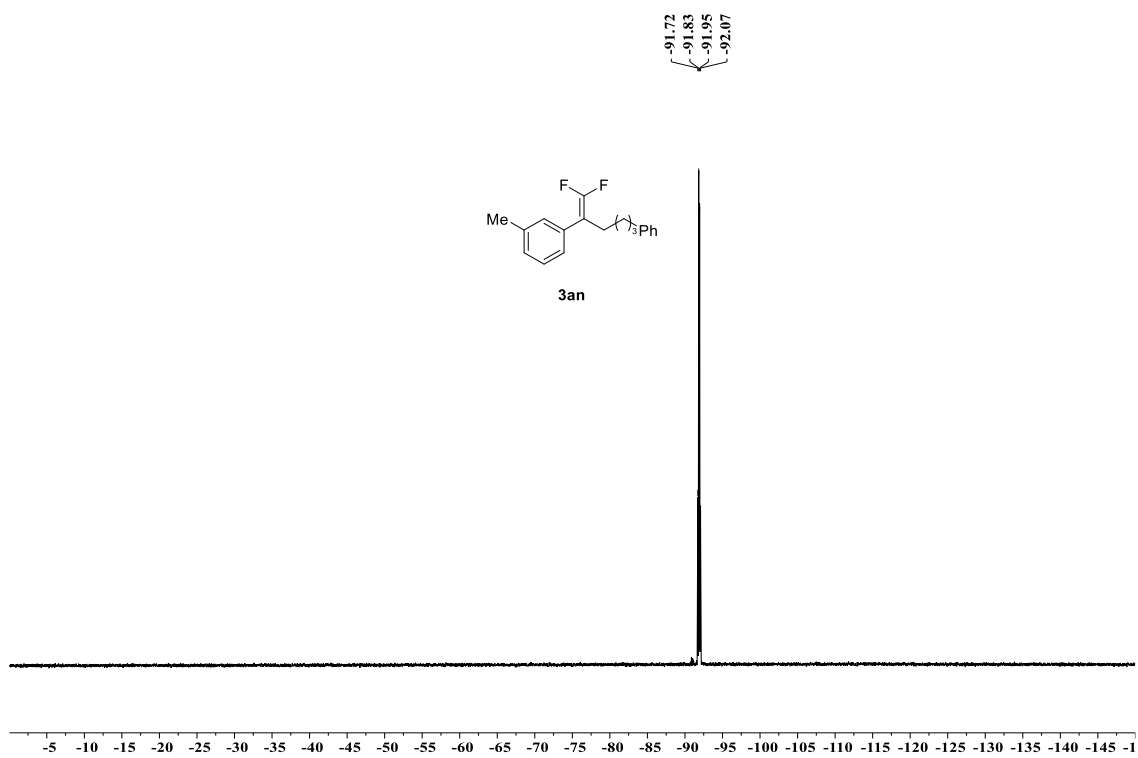
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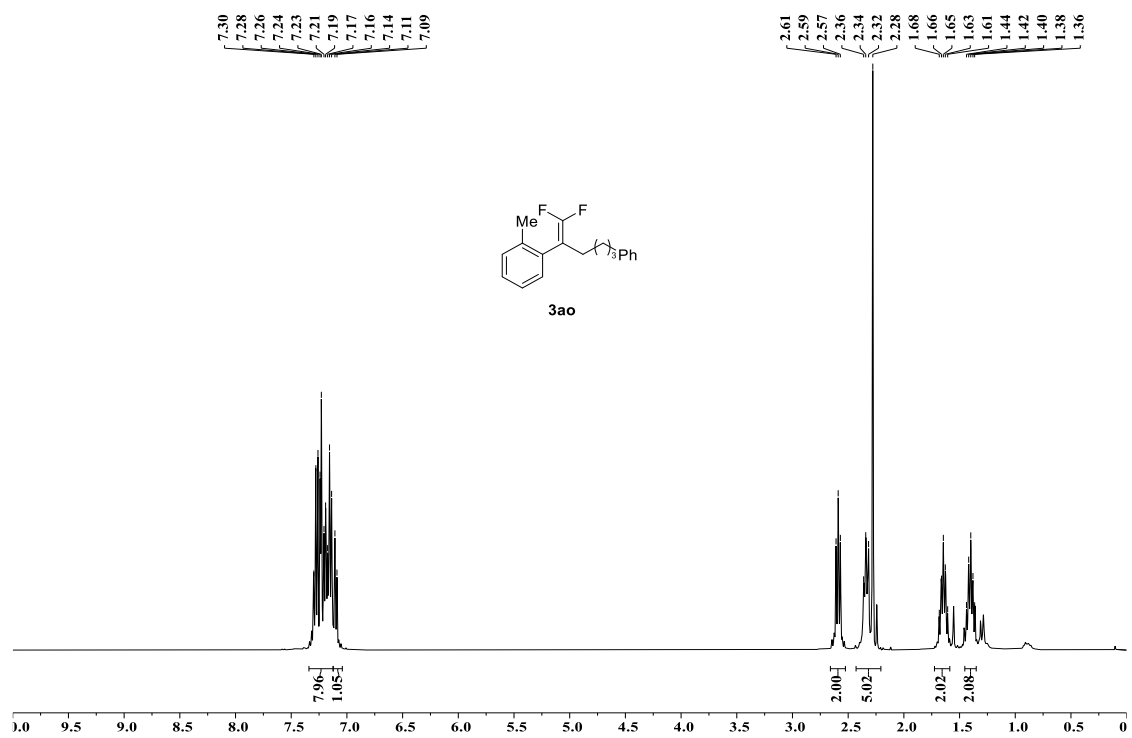
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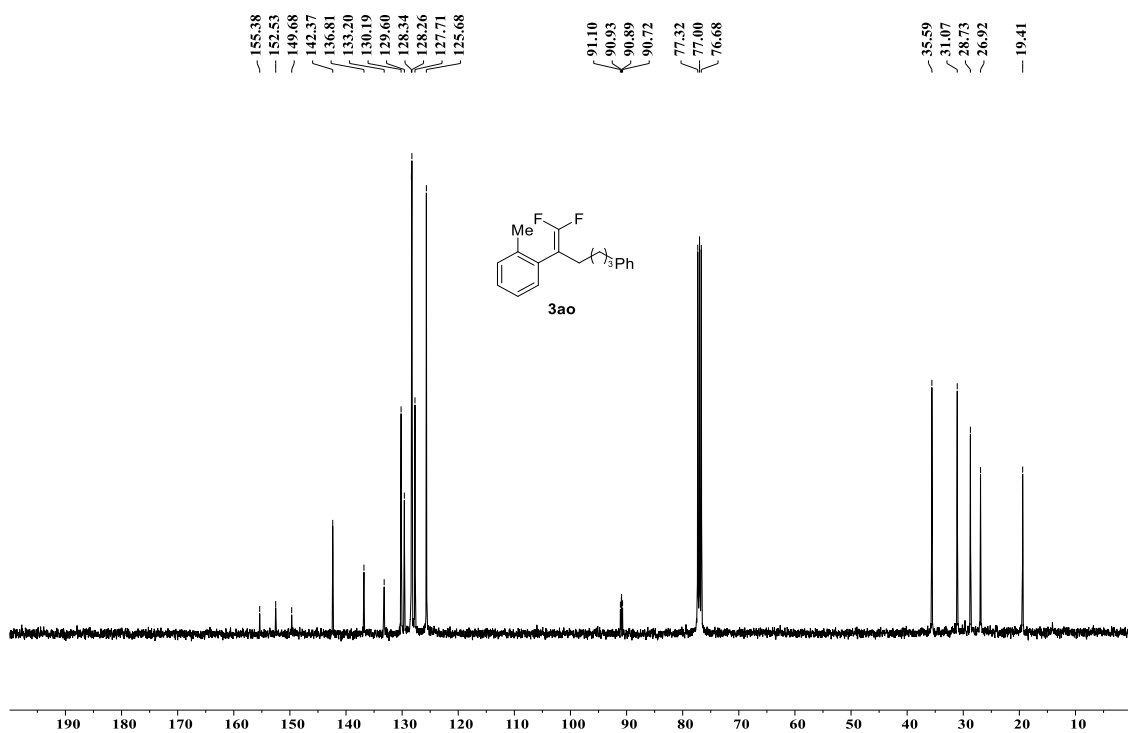
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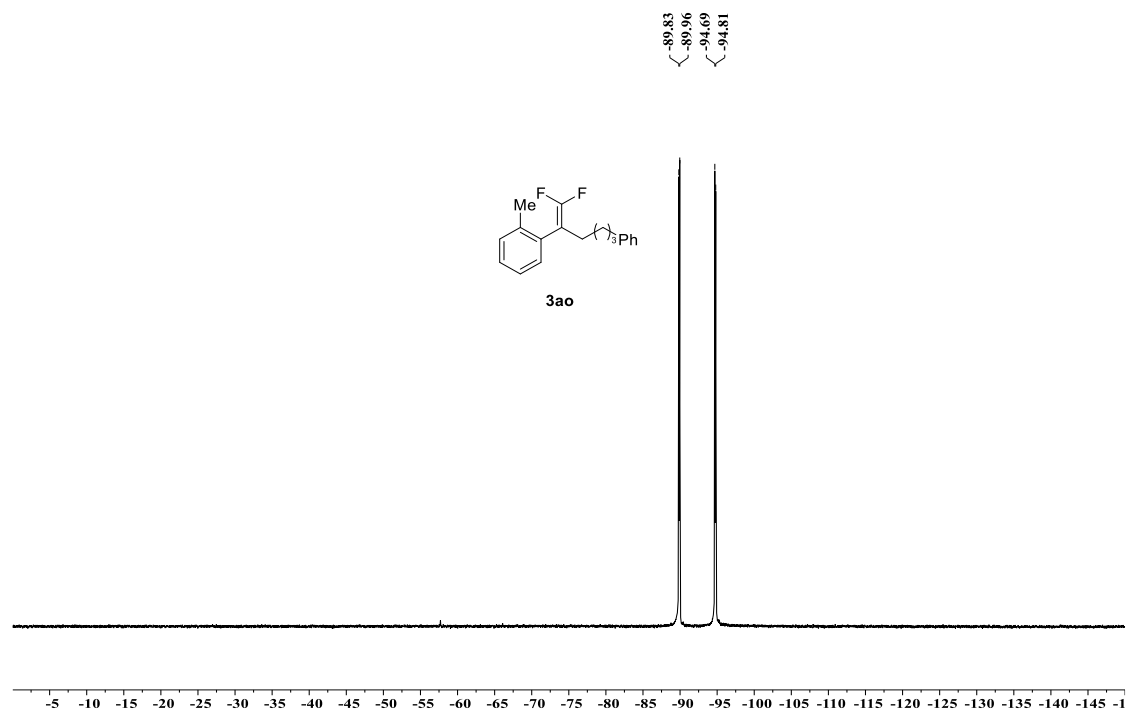
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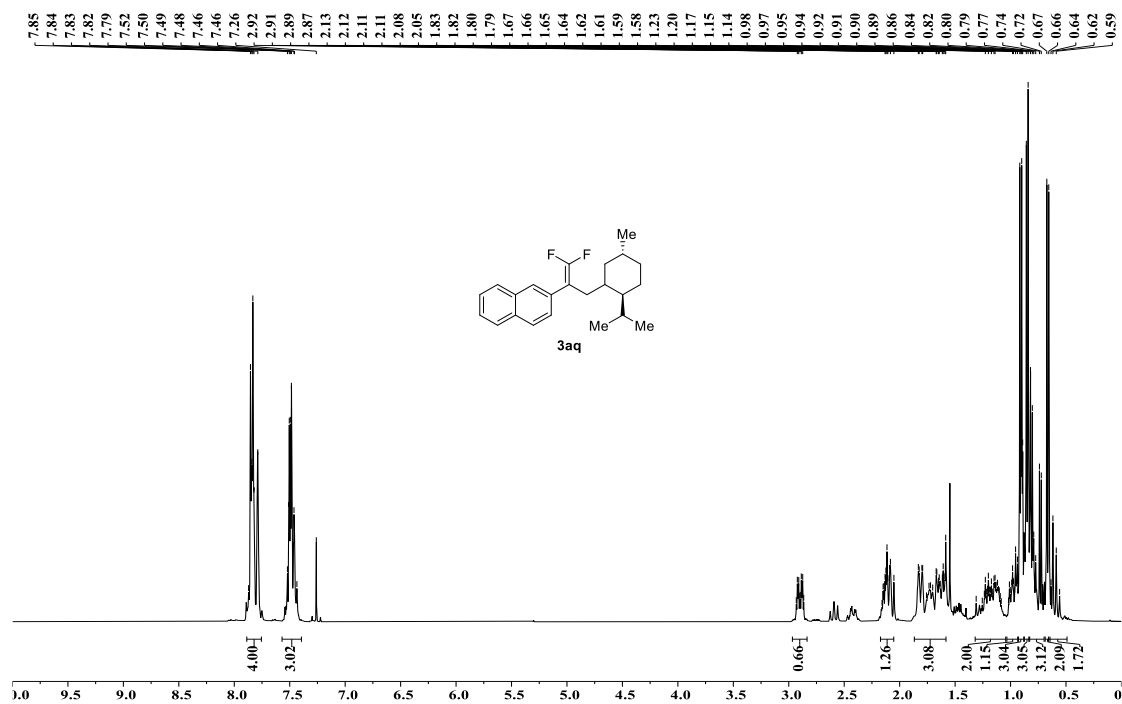
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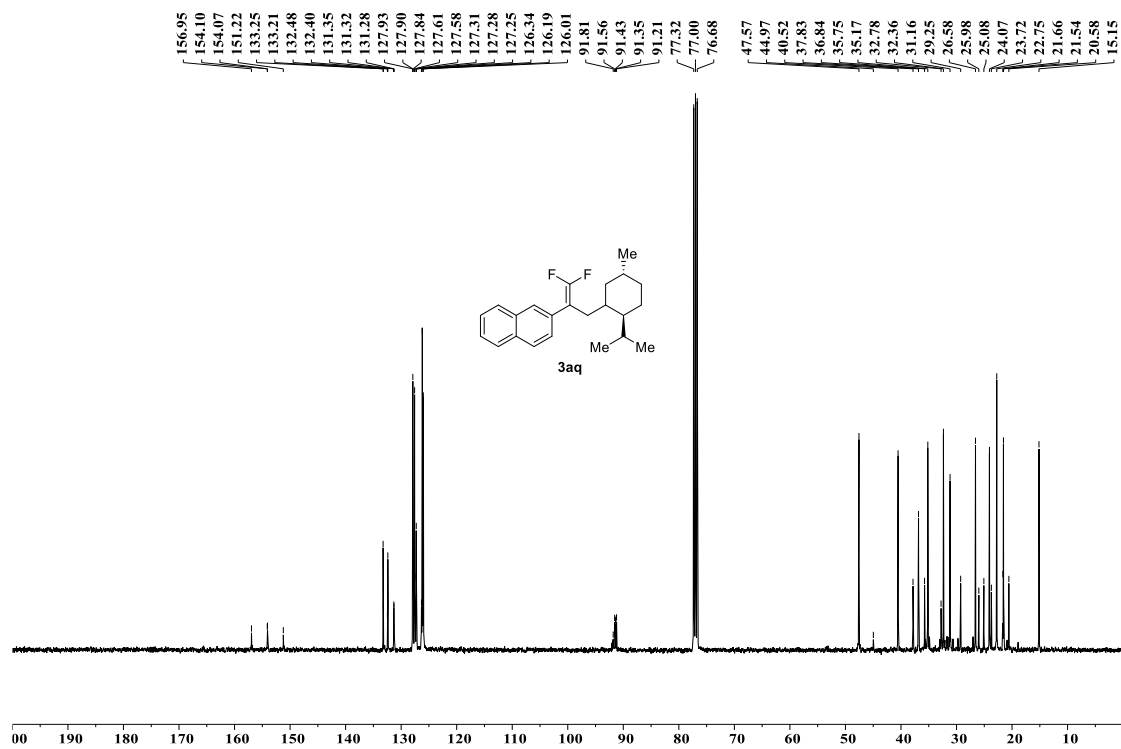
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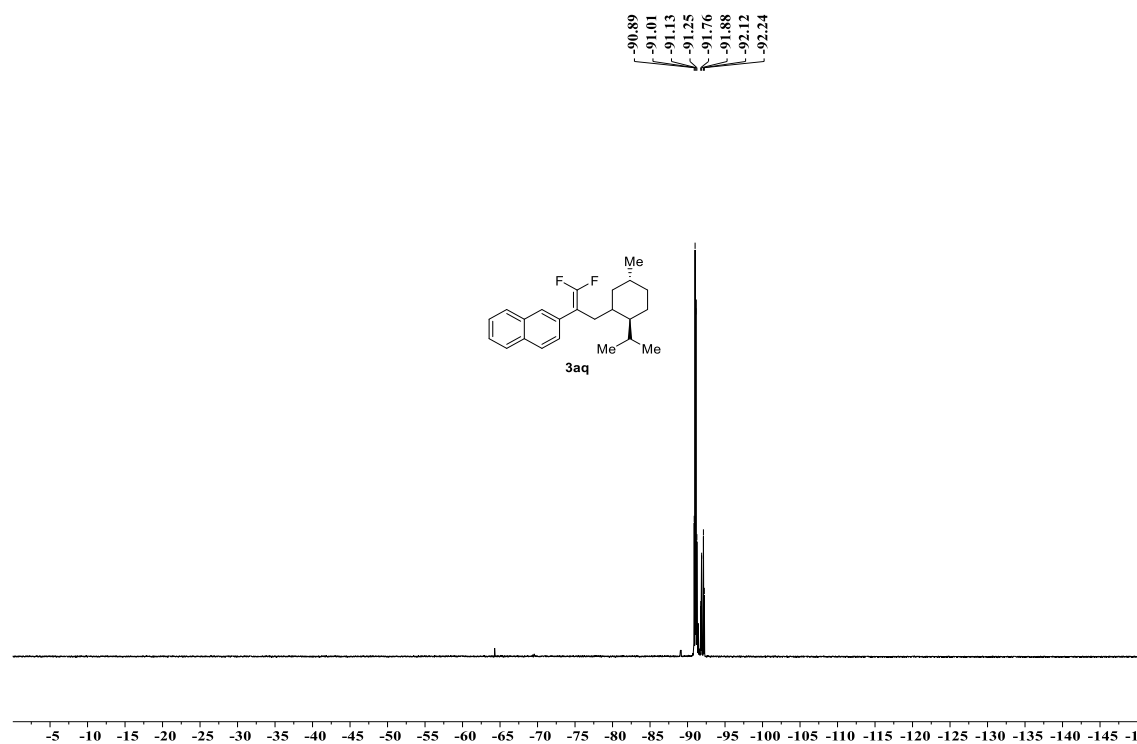
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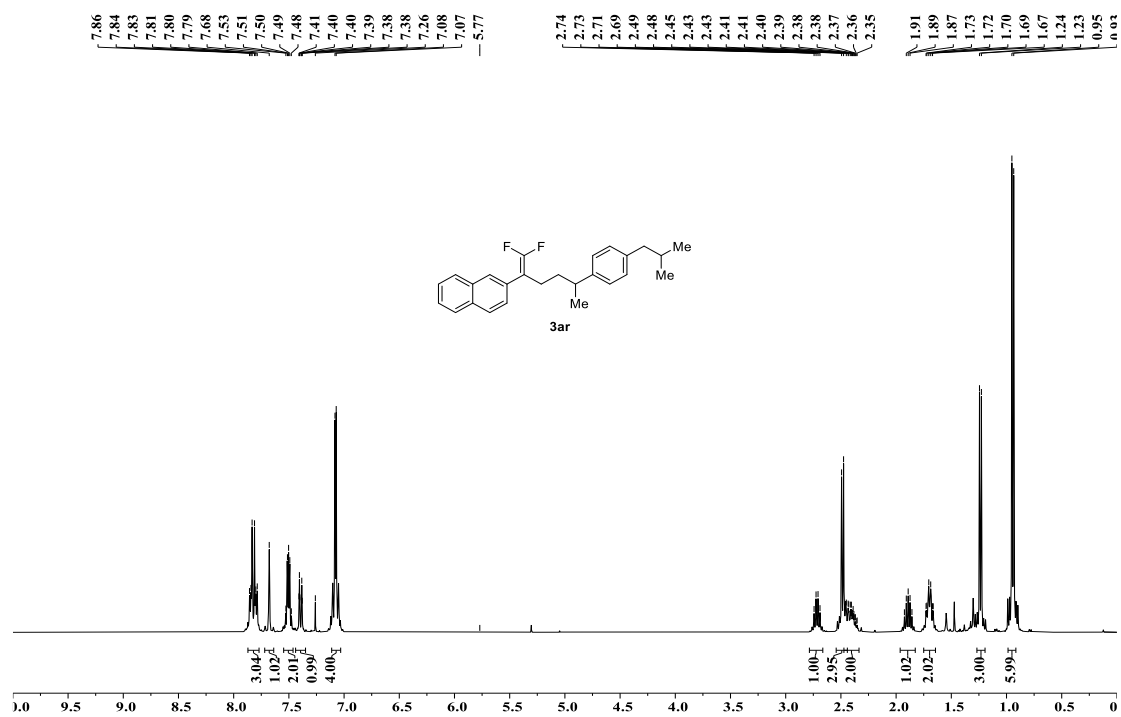
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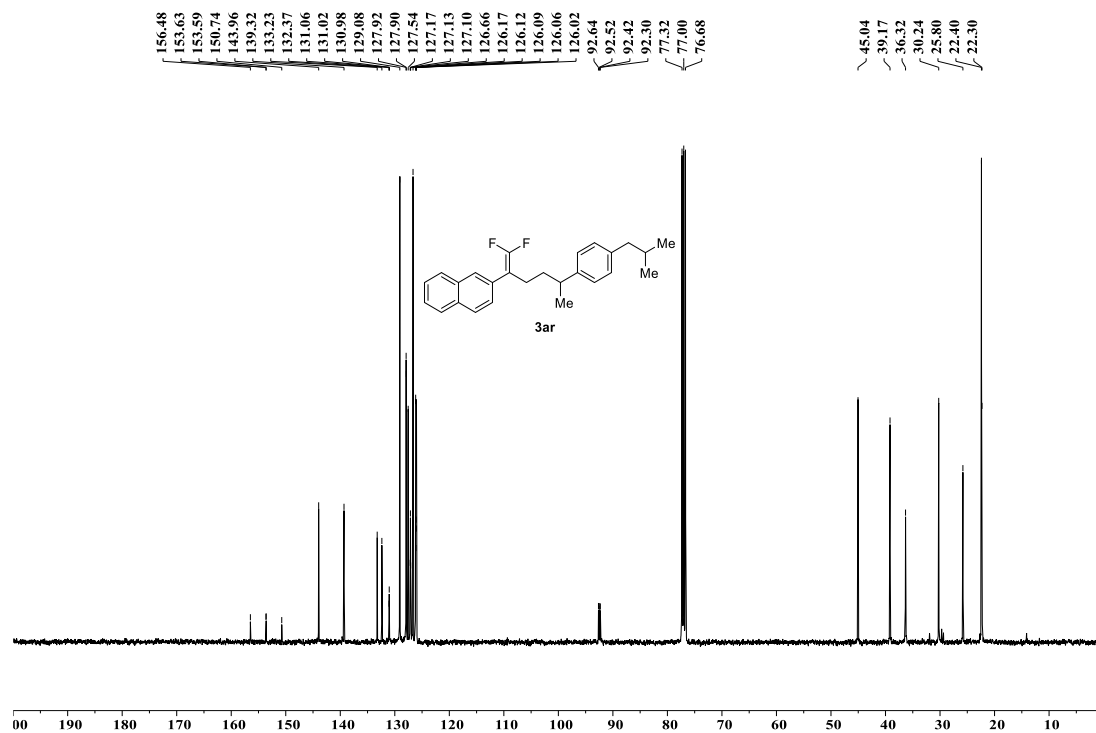
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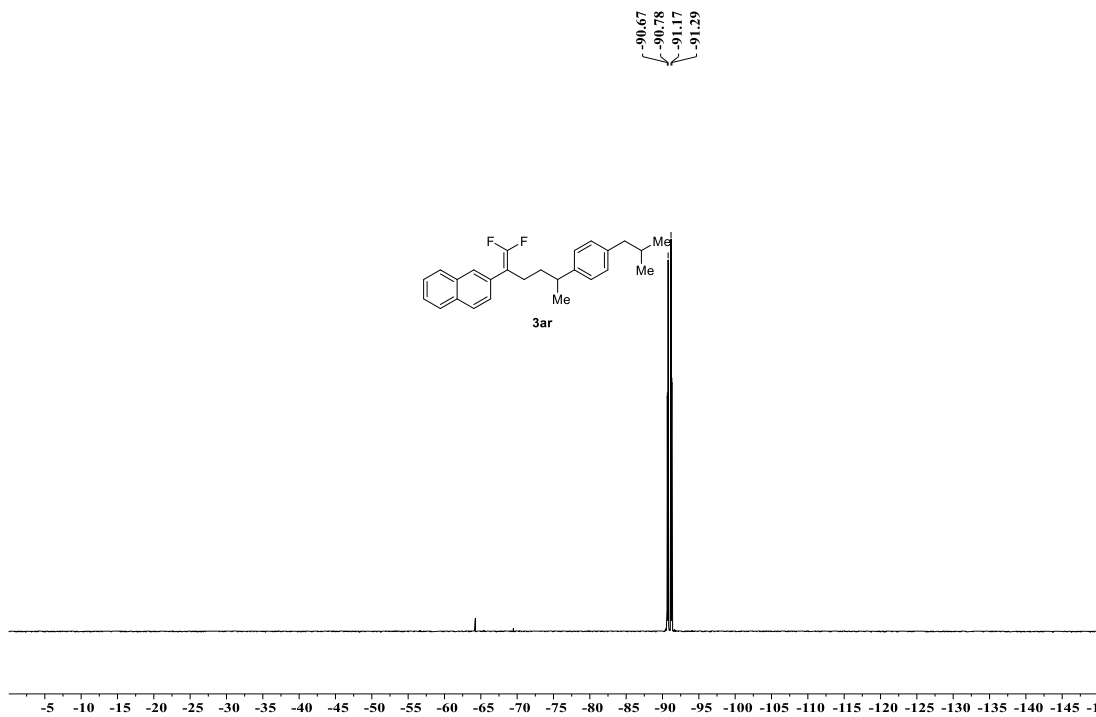
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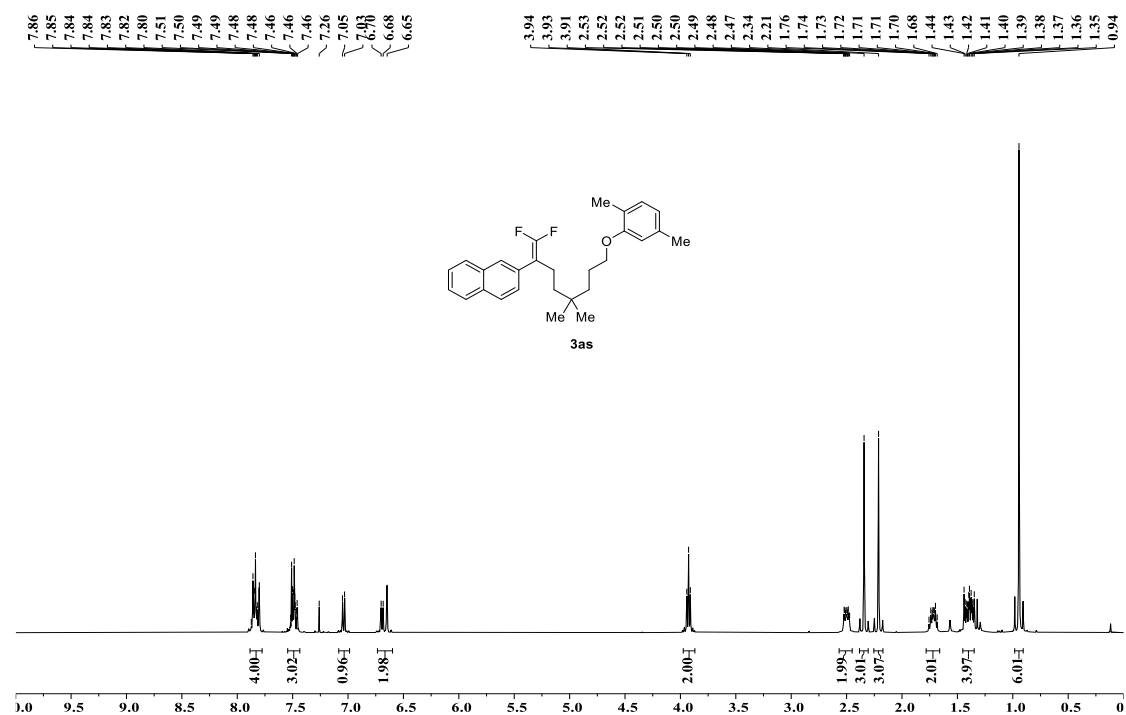
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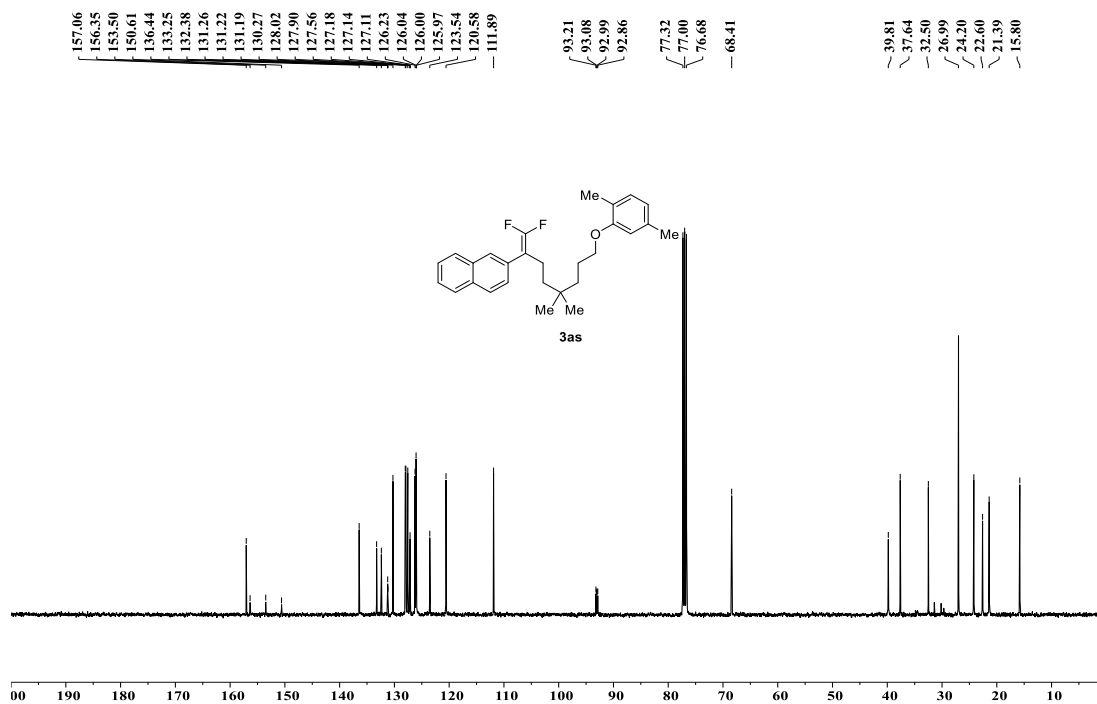
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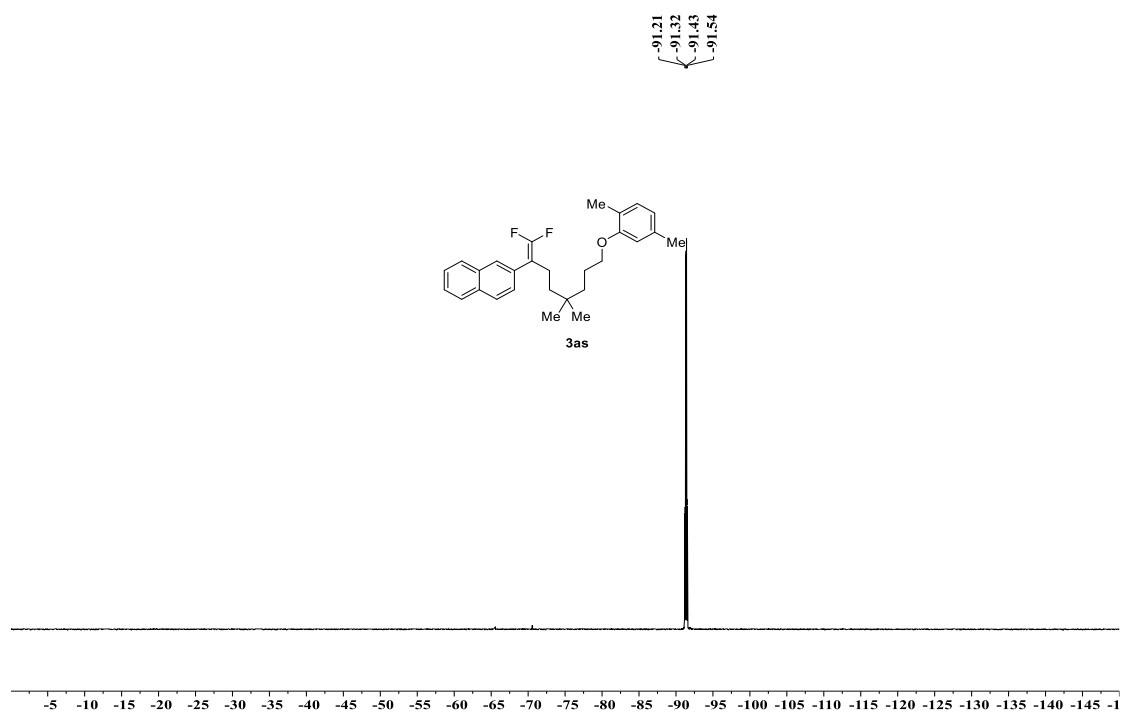
^1H NMR (400 MHz, CDCl_3)



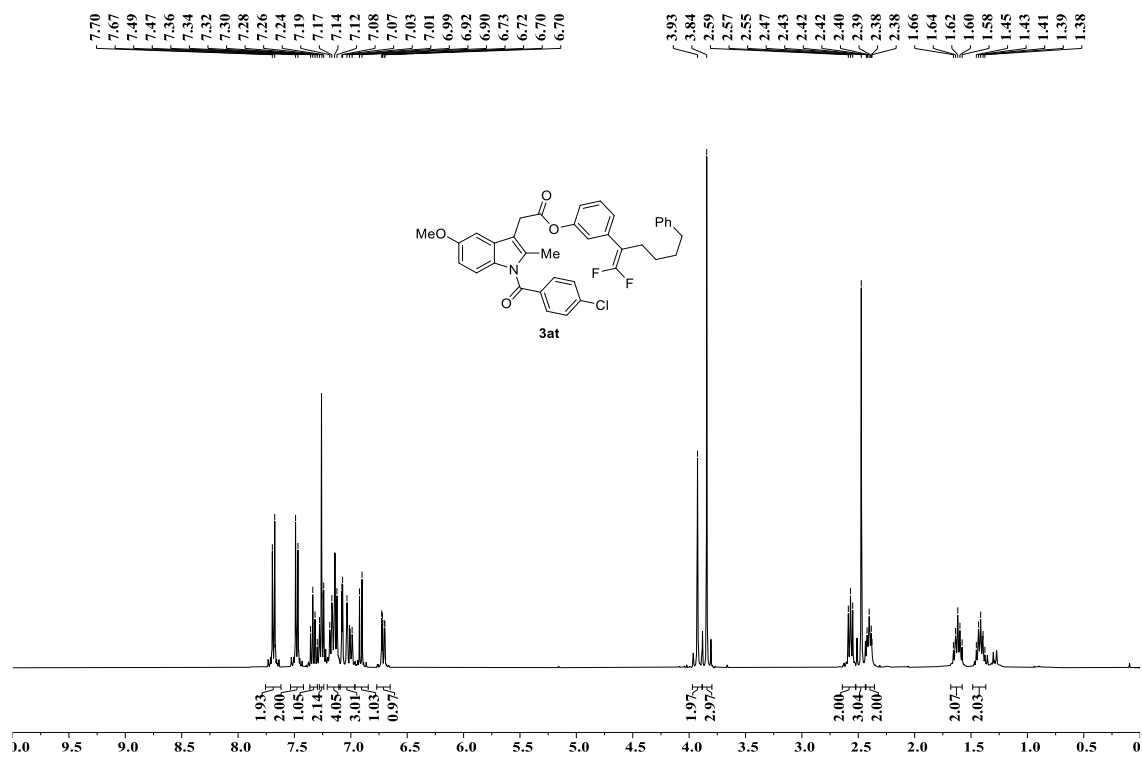
^{13}C NMR (100 MHz, CDCl_3)



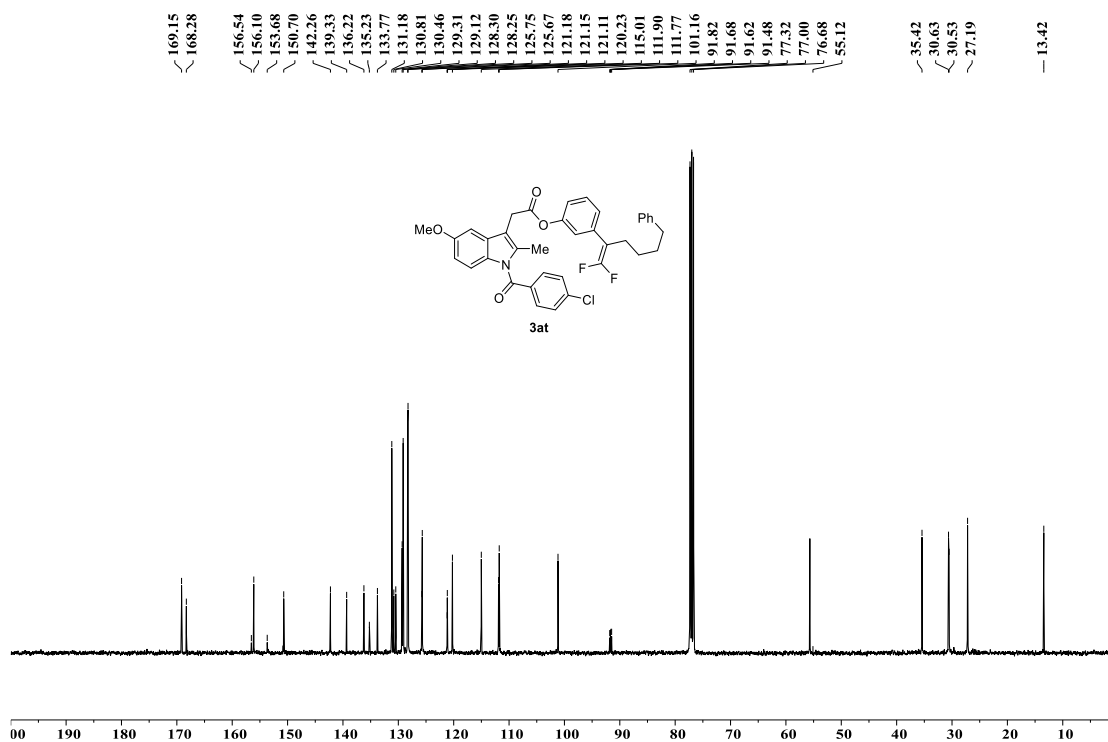
^{19}F NMR (376 MHz, CDCl_3)



^1H NMR (400 MHz, CDCl_3)



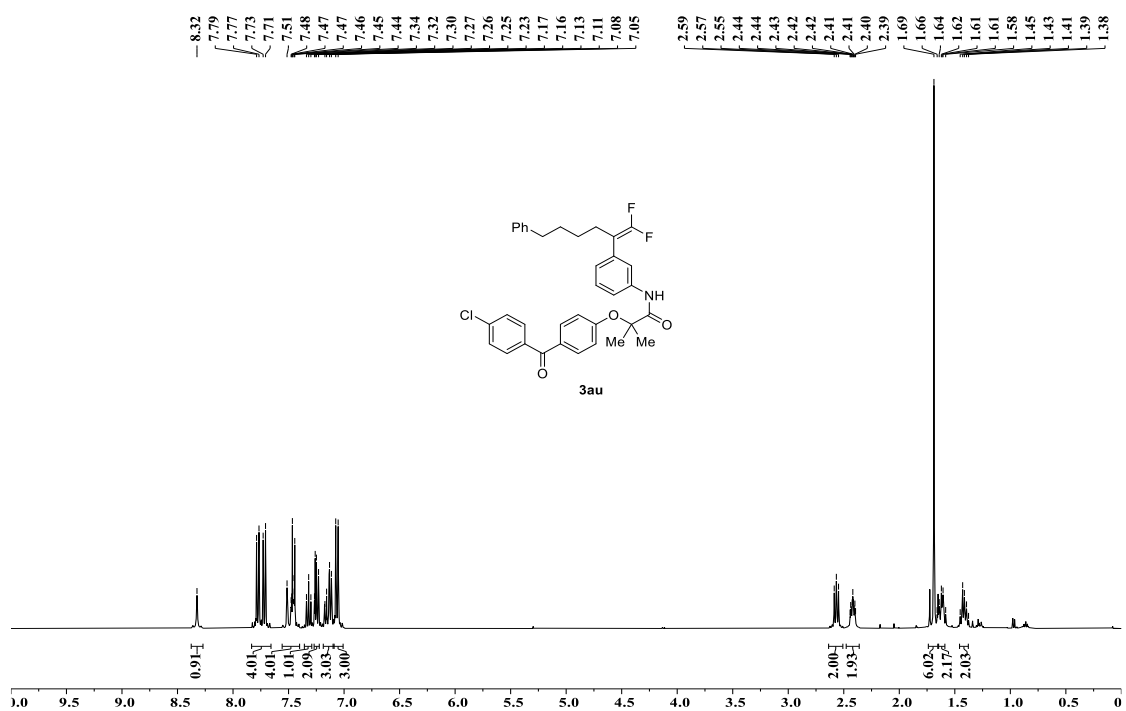
¹³C NMR (100 MHz, CDCl₃)



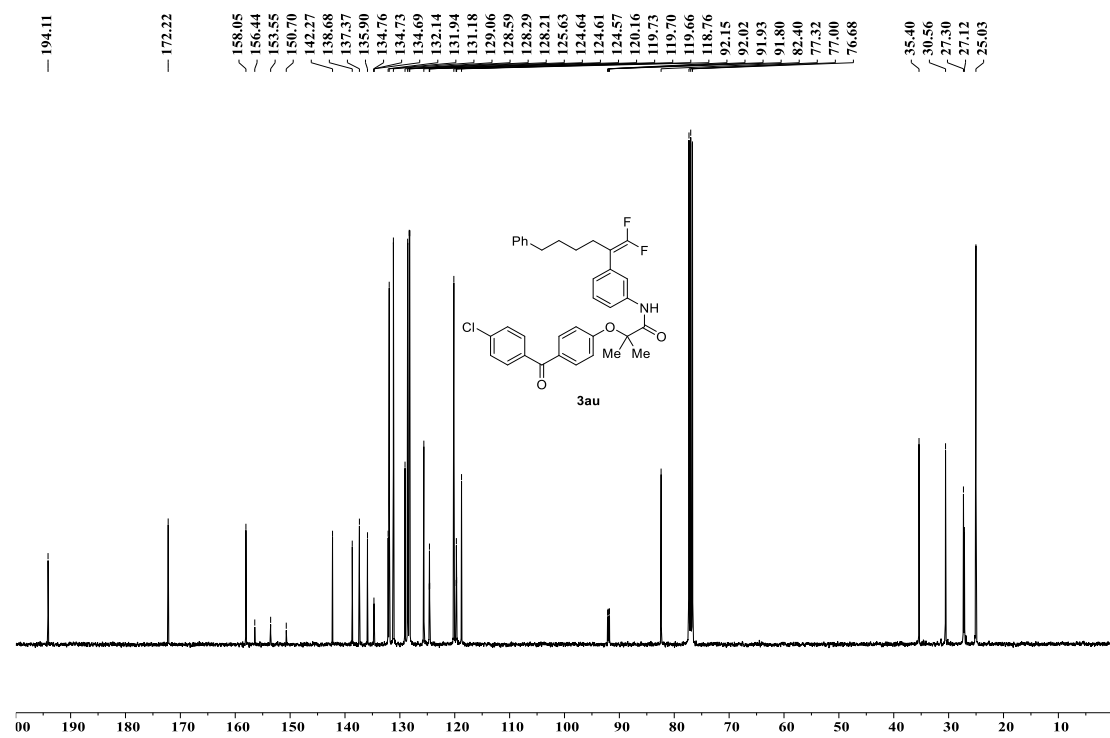
¹⁹F NMR (376 MHz, CDCl₃)



^1H NMR (400 MHz, CDCl_3)



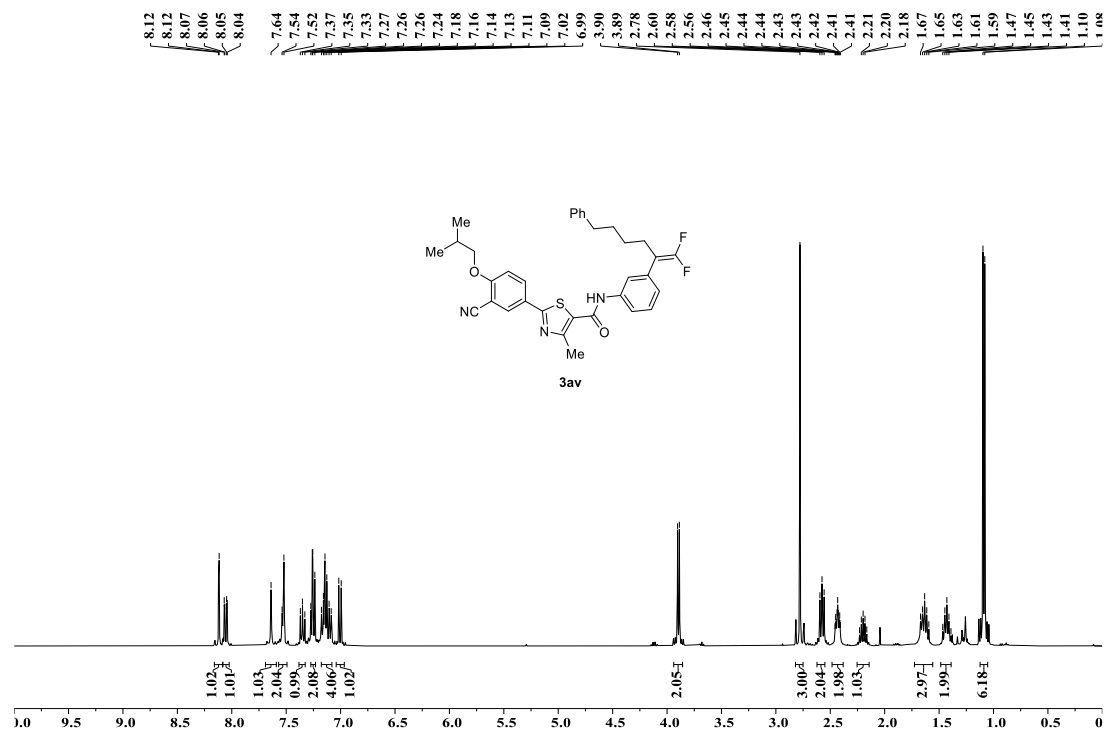
^{13}C NMR (100 MHz, CDCl_3)



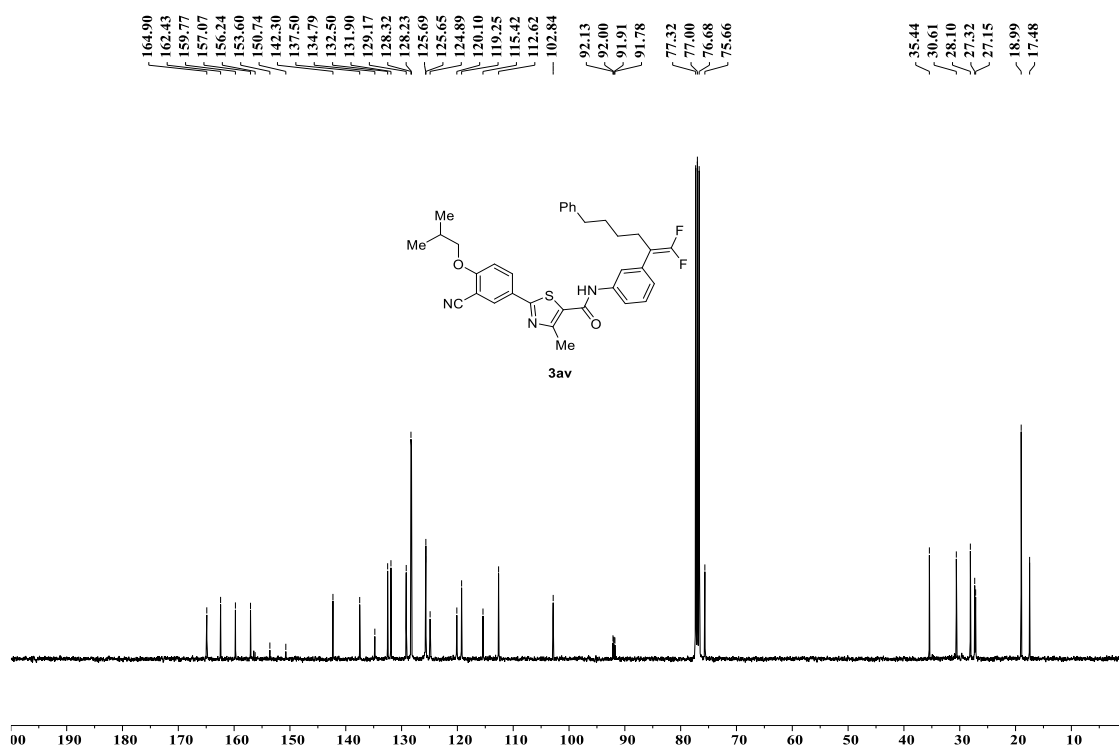
^{19}F NMR (376 MHz, CDCl_3)



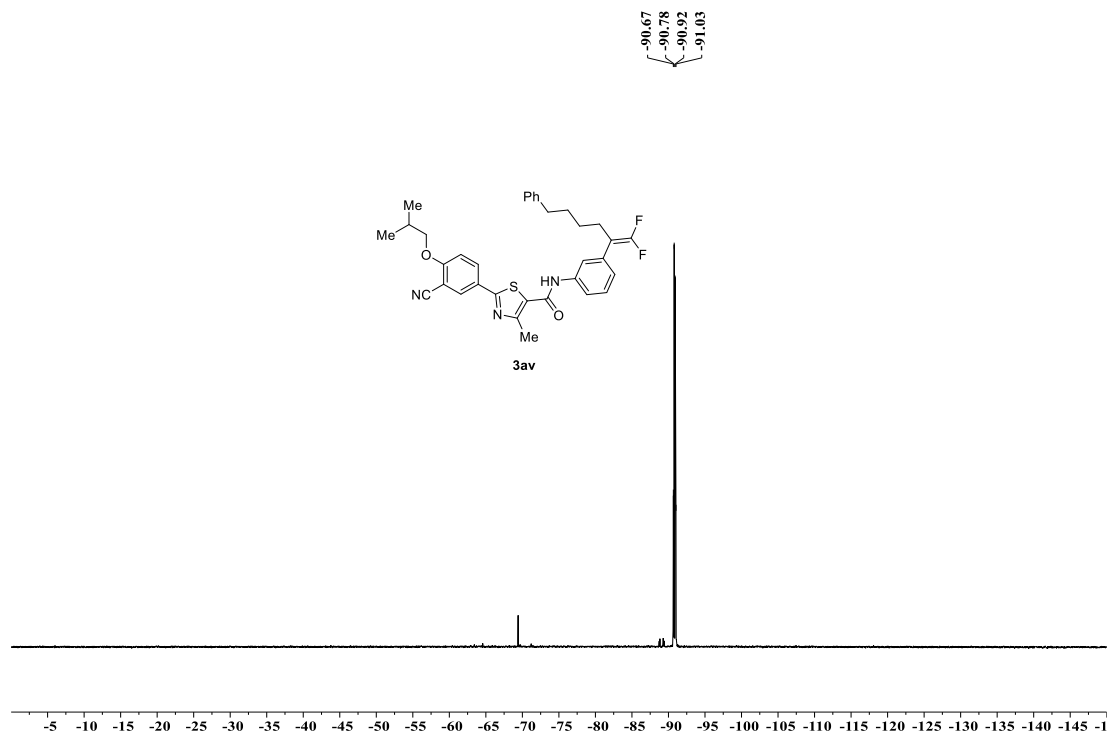
^1H NMR (400 MHz, CDCl_3)



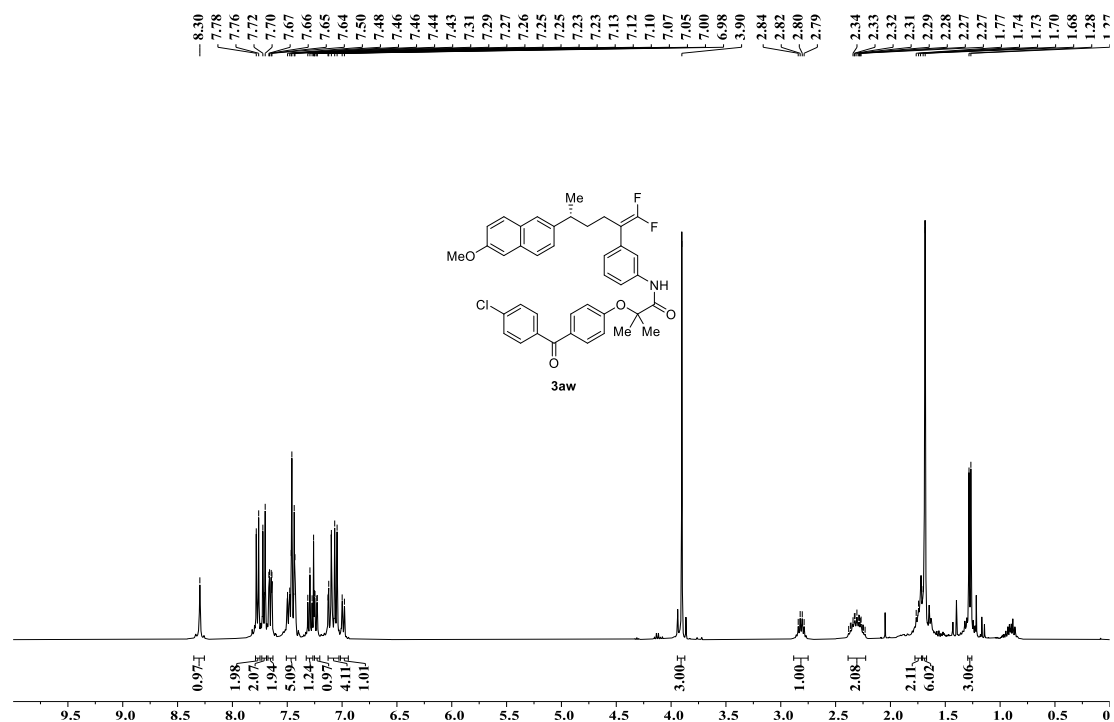
^{13}C NMR (100 MHz, CDCl_3)



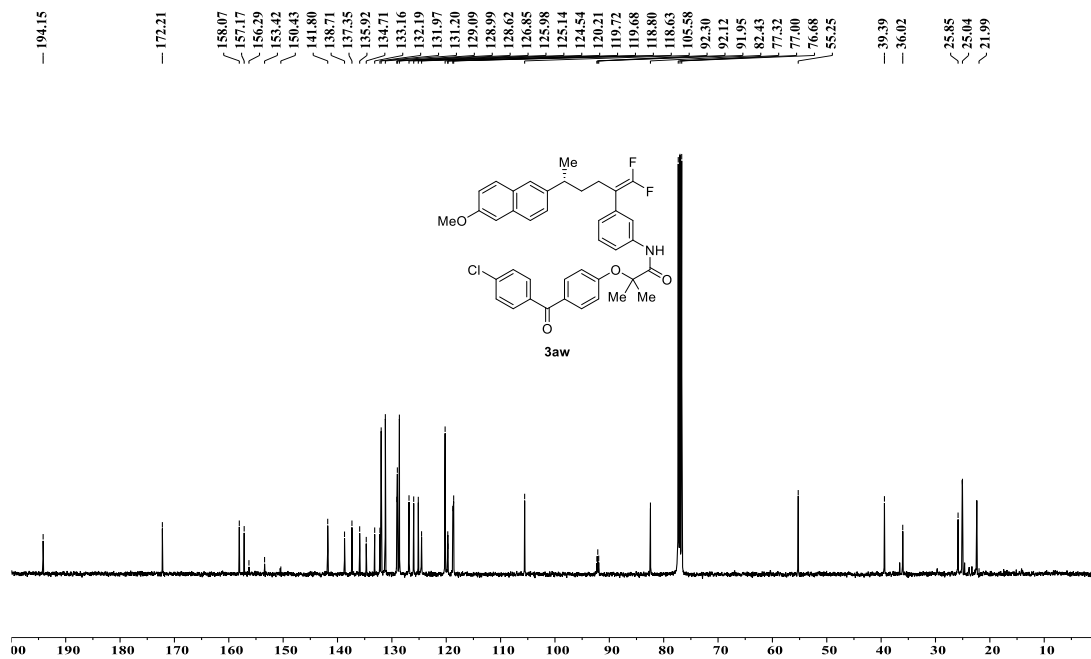
^{19}F NMR (376 MHz, CDCl_3)



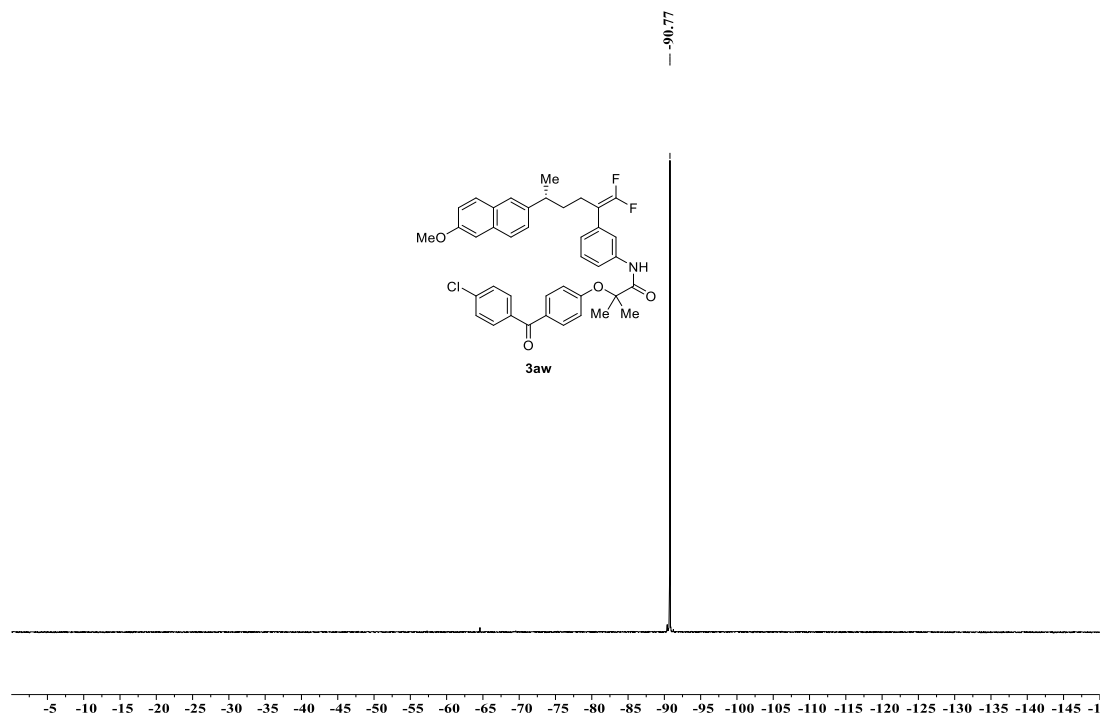
^1H NMR (400 MHz, CDCl_3)



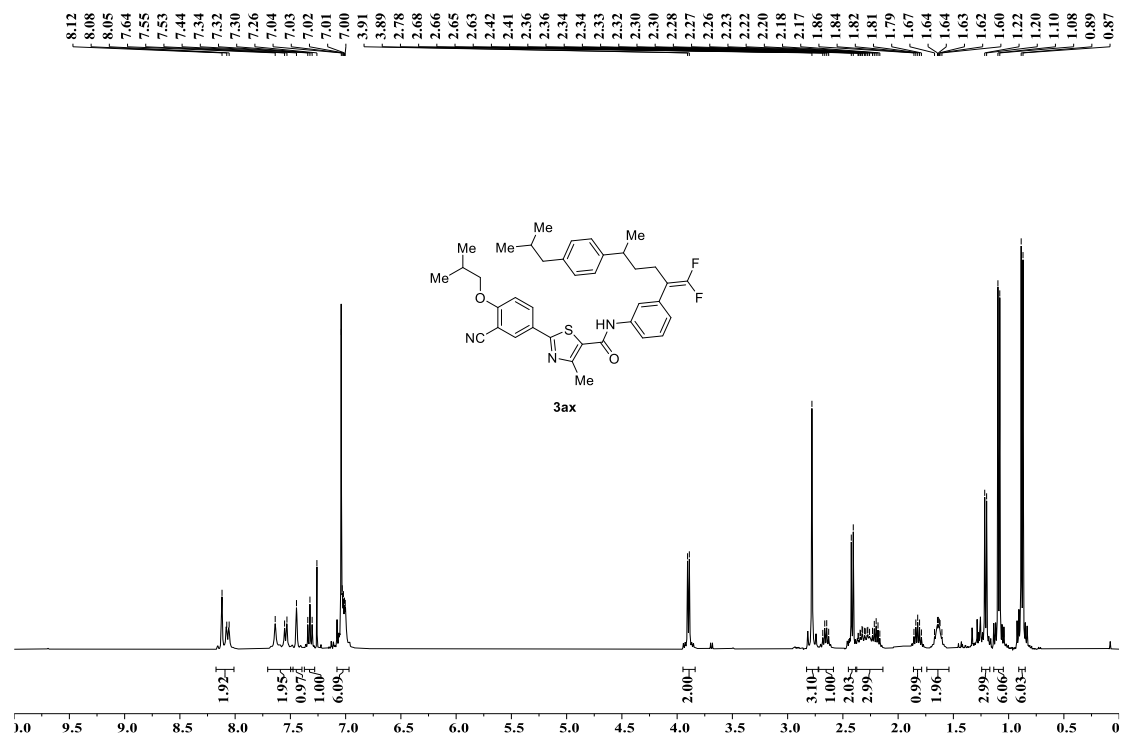
^{13}C NMR (100 MHz, CDCl_3)



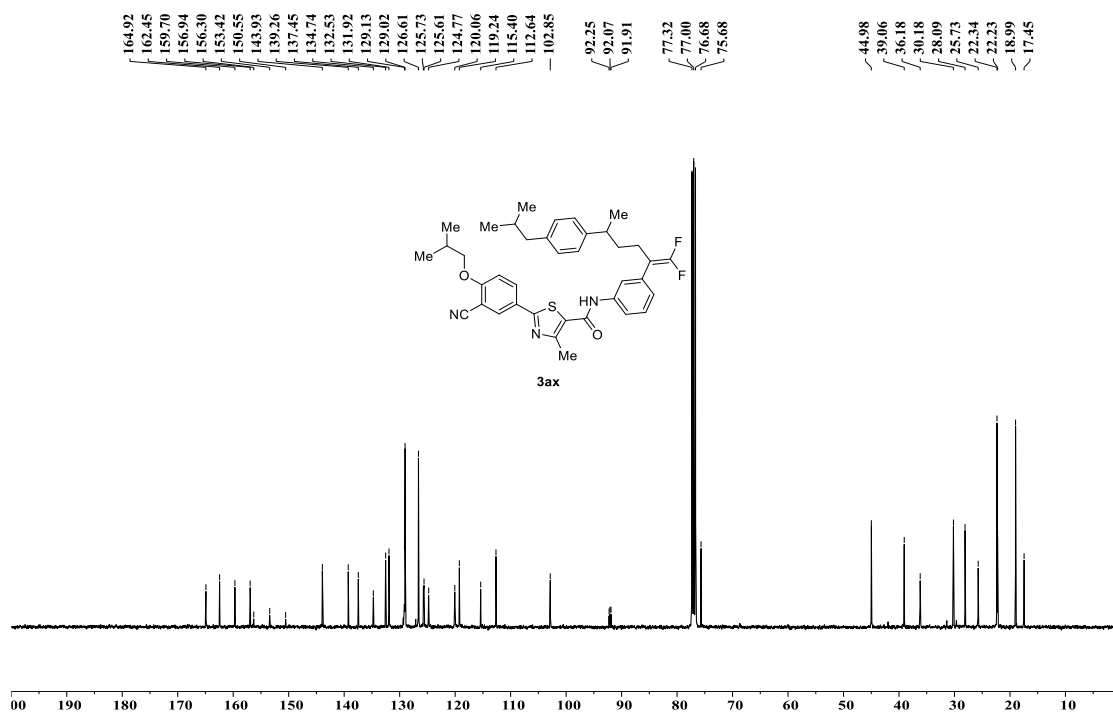
^{19}F NMR (376 MHz, CDCl_3)



^1H NMR (400 MHz, CDCl_3)



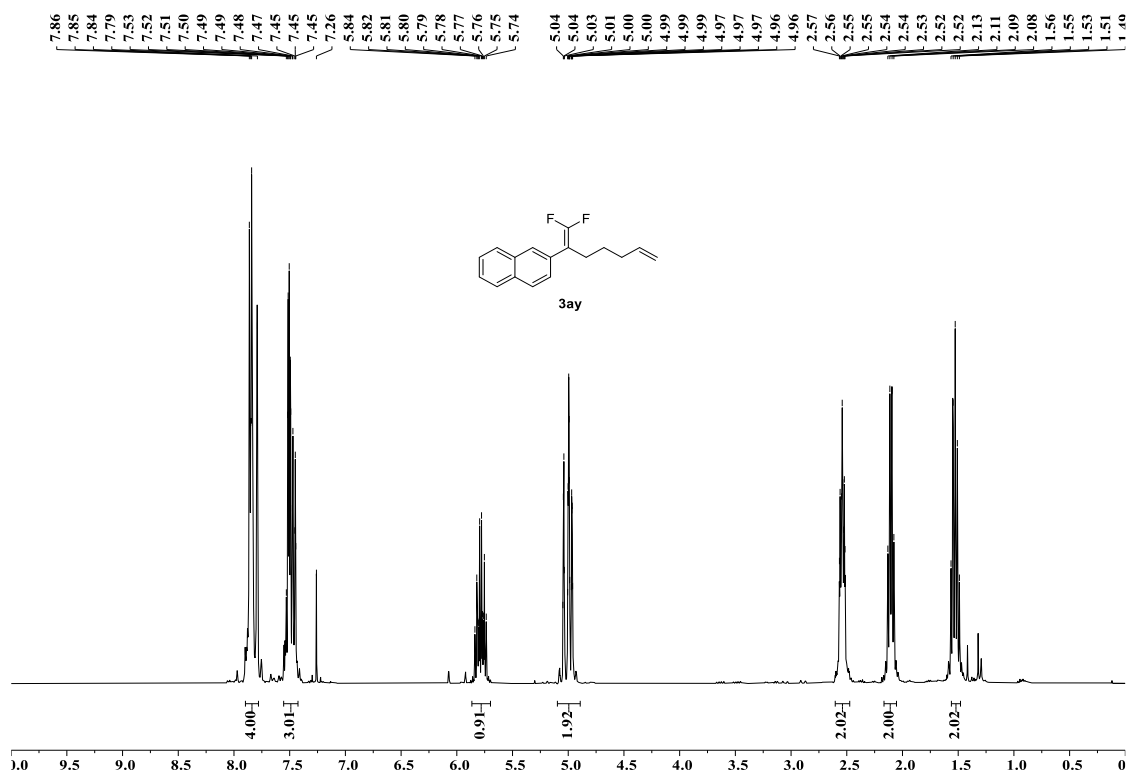
¹³C NMR (100 MHz, CDCl₃)



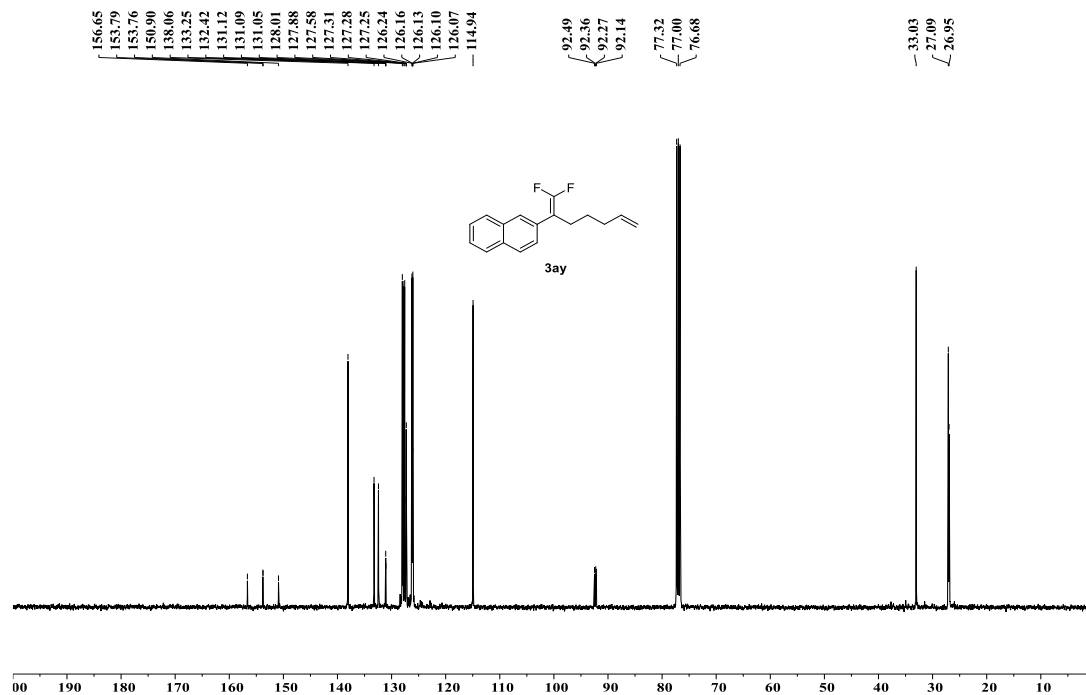
¹⁹F NMR (376 MHz, CDCl₃)



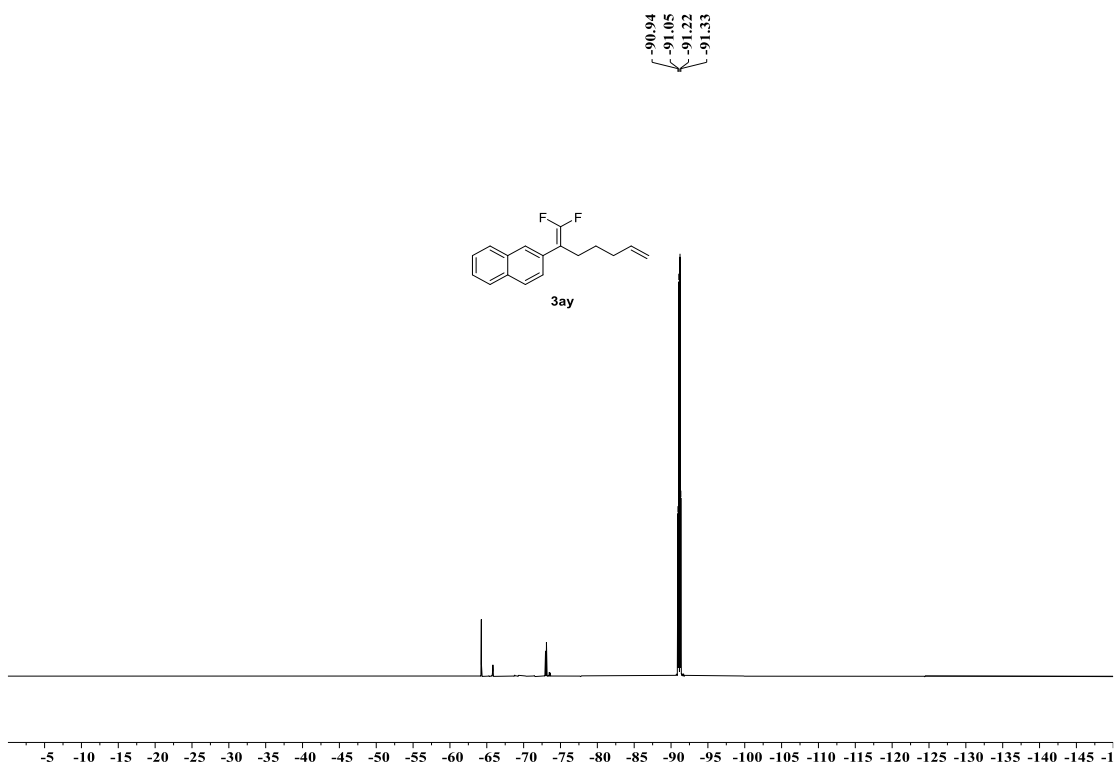
^1H NMR (400 MHz, CDCl_3)



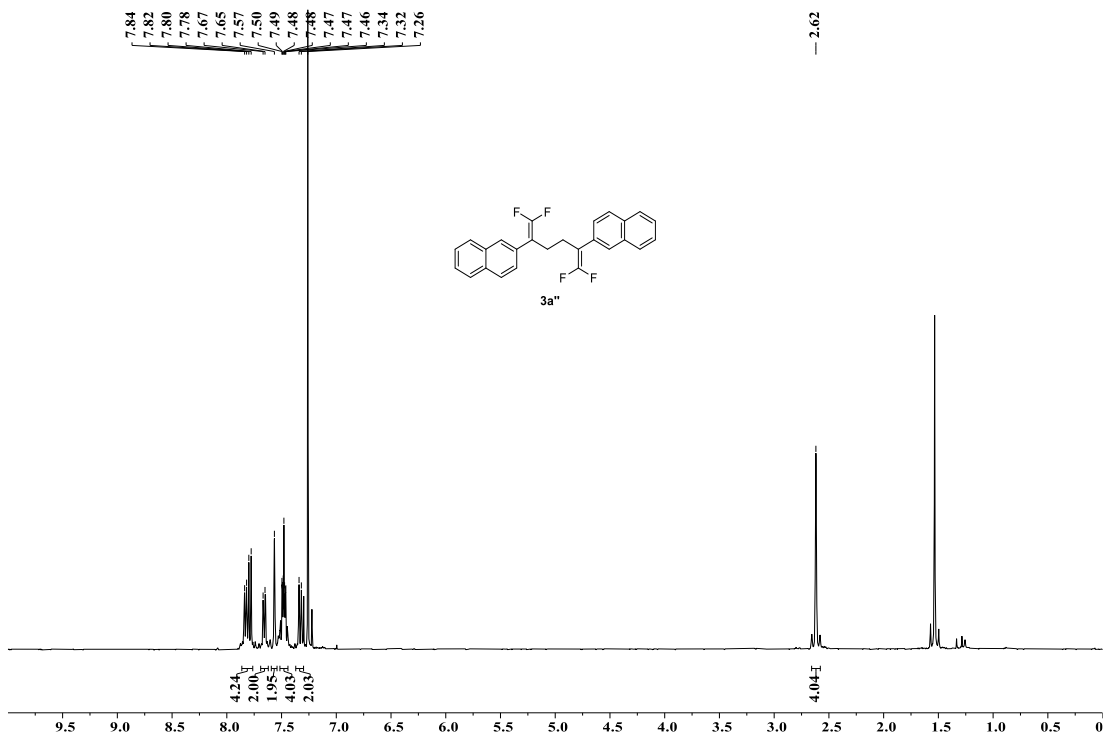
^{13}C NMR (100 MHz, CDCl_3)



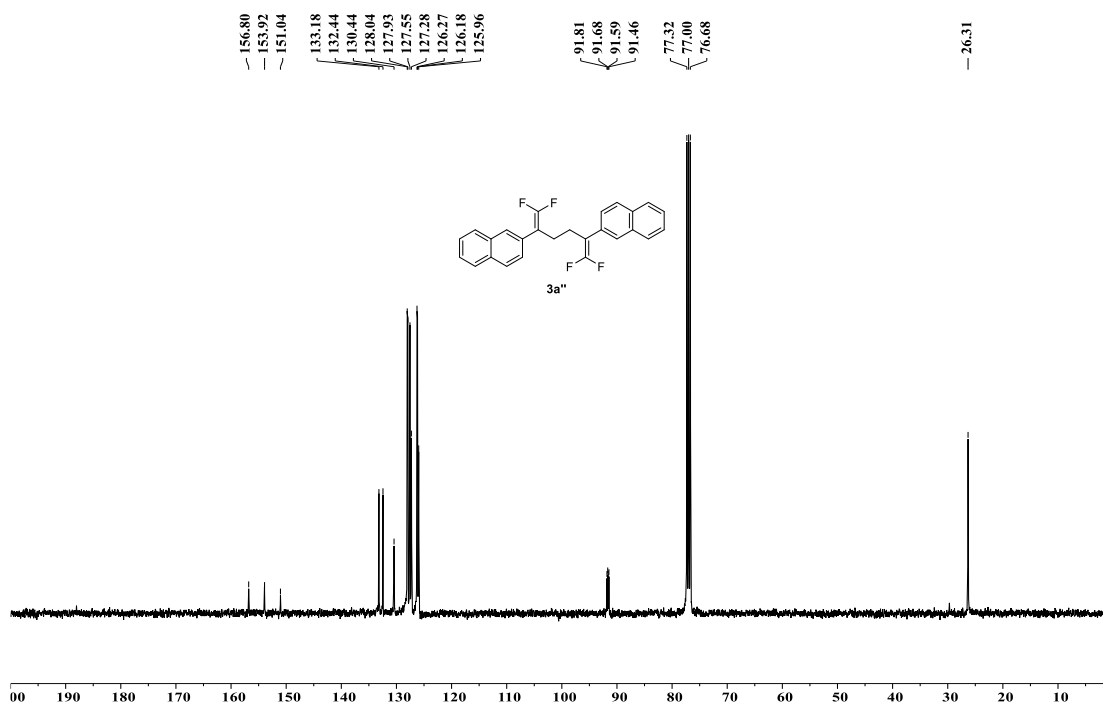
^{19}F NMR (376 MHz, CDCl_3)



^1H NMR (400 MHz, CDCl_3)



^{13}C NMR (100 MHz, CDCl_3)



^{19}F NMR (376 MHz, CDCl_3)

